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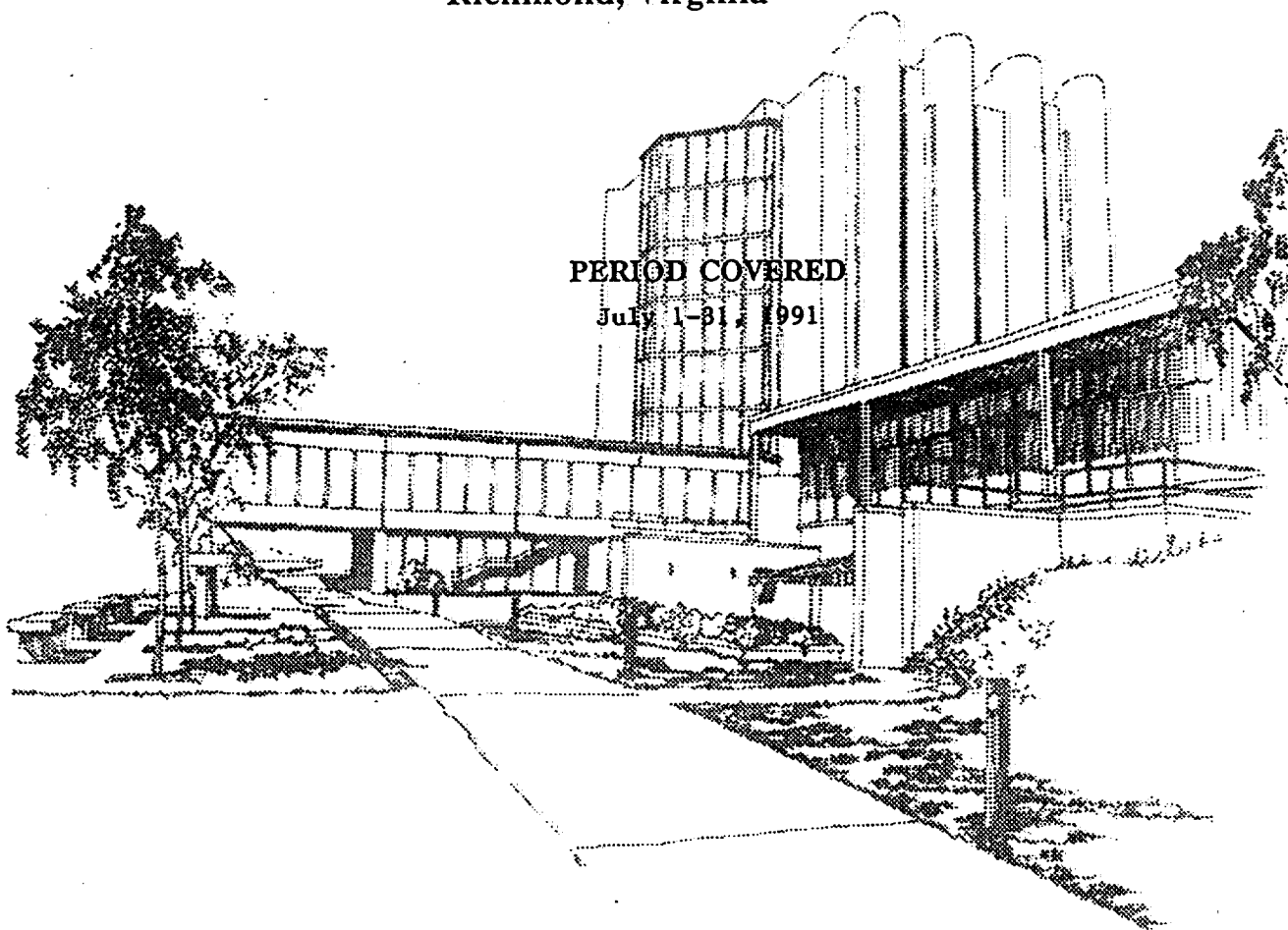
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**PHILIP MORRIS USA**  
Research Center  
Richmond, Virginia

PERIOD COVERED

July 1-31, 1991



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\* N/R = No Report

**PROJECT NUMBER :** 2108  
**PROJECT TITLE :** New Product and Filter Technology  
**SECTION LEADER :** W. T. Callahan  
**PERIOD COVERED :** July, 1991

## **I. FILTER WEB DEVELOPMENT**

- A. Objective:** To characterize web-type filter materials and develop subjectively acceptable products with them.
- B. Status:** Filters were produced with PM web made with 1, 2, and 3% CB-10 dispersion additive. Evaluation of filter physical quality and cigarette making indicated these materials to be typical of previous PM webs.

Cigarettes were produced with Courtaulds partially de-acetylated cellulose acetate web. The models will be subjectively evaluated against a Control.

PM Web materials produced during the week of June 25 at James River Corporation's Neenah Technical Center were used for filter rodmaking and testing. These materials include cellulon, cotton, and fibrids along with CA staple. The runnability of the CA/Cellulon and CA/CA Fibrids was poor due to poor corrugatability. Inclusion of 10% softwood in the CA/Cellulon web improved the runnability. CA/Cotton was very sensitive to corrugation stress, however, the material did machine acceptably. Variability data is being analyzed currently.

Specifications were written and forwarded to PM engineering for the production of web filter rods by Decoufle, a French machine manufacturer. The filters will be analyzed for variability and fitness for use on PM prototype products.

A bobbin of the latest CA web was received from Celanese. The material was produced in March, 1991, by Kimberly-Clark. The material will be evaluated and compared to data from previous CA web material evaluations in order to assess any manufacturing and/or filter-making improvements which may have occurred.

## **II. MACHINERY DEVELOPMENT**

- A. Objective:** Develop or modify manufacturing equipment to support Product Development efforts.
- B. Status:** Initial attribute testing by QE of eight test filters produced on the Hauni ACC P-S-P combiner indicated significant carbon scatter. Cigarettes made from these models will begin subjective testing in 1-2 weeks.

Modifications are continuing to be made to the Strip Application Unit as individual problems are identified during testing. The cork drum was redesigned and tested successfully and currently the cutter head is being modified.

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A meeting was held with Molins ATU personnel to review the design of their proof-of-concept rig. Operating and product specifications were developed with Engineering and Project Tomorrow personnel. Specifications for lap seam adhesive and a video tape of the PM Strip Application Unit in operation were transmitted to aid in their development. Debugging trials will begin at Molins about August 1. Bobbins of standard cigarette wrapper have been slit to 23mm width and shipped for these trials.

More drawings for the laboratory coater have been received from Fox Valley Ltd. Some design changes to assure consistent web tension have been requested of them. A visit was made to Fox Valley to review the design and observe a similar machine built for Kimberly-Clark (currently in their shop).

### III. NOVEL FILTERS AND FILTER MATERIALS

- A. **Objective:** To investigate novel filters or filter materials for potential applications where increased efficiency, taste response or a novelty is needed.
- B. **Status:** BOLD 100's cigarettes produced with James River Gouverneur Mill paper cores were evaluated by the Richmond Panel against a control model and achieved subjective parity. A larger run of this material is planned to support POL testing.

The BOLD factory trial at Cabarrus indicated no problems with King Size filter APHIS feeding, tipping or filter machinability. In the 100s circumference study, all filters in the range of 24.1mm to 24.8mm machined acceptably and passed audit testing by R&D and QA.

A ripper room trial was conducted with the PCC filters and Engineering considers the Cabarrus system acceptable for this filter. Additional optimization is required prior to startup.

BOLD filters continue to be received, sampled and inspected to verify adherence to the lot acceptance plan. Regular weekly meetings are being held with representatives of American Filtrona to review the progress of production of BOLD filters.

Cigarettes were smoked on standard and dual Cambridge pads to provide TPM for fractionation studies by F. Hsu.

Cambridge Lowest 100s cigarettes were made with 1.2 dpf Eastman tow and tested analytically and physically. The control model was the current production design using 1.8/35,000 tow. The results indicate a 22% reduction in tar on the 1.2 dpf filter at equal filter RTD and ventilation. Using design model data, a ventilation decrease of approximately 8% on the test model would yield a tar delivery equal to the control. Additional filters have been requested from Eastman for more extensive testing.

Meetings were held with representatives from Ecusta and Kimberly-Clark Corporations to review data from recent development trials. KC subsequently produced additional models which were run in a factory trial on Lark Milds KS at

the Manufacturing Center. The two models were at two levels of porosity and were run on three different tipping glue rollers. Ventilation levels of the models are being determined and cigarettes have been submitted to MC Q.A. for defect analyses.

Work has begun on a second run of Parliaments using spiral wound tubes as the mouthpiece to provide the recess.

Cigarettes with hand attached impaction filters and hand ventilated to 30% and 50% levels were machine smoked versus a control Half-Nic model. These models were smoked by the Flavor Technology Domestic Product panel.

Filters were sampled and tested in support of production of IM #14 monitor cigarettes and were analyzed as being physically acceptable.

#### IV. ALTERNATIVE PLASTICIZERS

- A. **Objective:** Qualify alternative plasticizers which offer a product advantage.
- B. **Status:** Marlboro KS regular cigarettes with Triacetin and Triethyl Citrate plasticized filters were subjectively evaluated by the Richmond Panel. The models were judged to be significantly different with the Triethyl Citrate model being mouth coating, bitter and dry at 12% ventilation and only mouth coating at 5% ventilation. Selected samples have been sent to ARD for smoke chemistry analyses to determine assignable causes for the subjective differences.

Marlboro Lights KS Menthol models with Triacetin and Triethyl Citrate plasticized filters were subjectively evaluated by the Richmond Panel. The models were judged to be significantly different with the Triethyl Citrate model being mouth coating. Mixtures of Triacetin and Triethyl Citrate will be investigated to eliminate the negative subjective characteristics.

#### V. CARBON STUDIES

- A. **Objective:** Conduct fundamental studies of carbons and carbon filtered cigarettes to better understand their performance.
- B. **Status:**

**Consolidation:** The Richmond panel found no differences between Lark Mild cigarettes made with standard production SCCW and Lark Mild cigarettes made with SCCW without zinc and iron additives.

Parliament 100's filters with Calgon MF2C carbon and with Calgon "PM Carbon" were produced in Semiworks for use in cigarette making at Stockton Street.

**Specifications:** A request has been submitted to Incoming QA in Richmond and Cabarrus for sieve size analyses of the three production carbons to characterize them using a new standard set of sieve screens. The standard set of sieve screens will be used by all PM USA carbon vendors for reporting sieve analyses and by QA when

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checking production carbons. The standard set of sieve screens include sieves #14, #20, #30, #40, #70, #100, and bottom pan.

Data was analyzed to determine the correlation of carbon tetrachloride activity with dry apparent density and with iodine adsorption. Iodine adsorption is not a feasible approach, but, approximately 75% of  $\text{CCl}_4$  activity variation can be explained by the apparent density. This approach is being evaluated to determine its usefulness as a valid QA testing procedure for activity.

## VI. MENTHOL TECHNOLOGY

- A. **Objective:** To develop alternative mentholation techniques which offer a product advantage.
- B. **Status:** Cigarettes with filters made with menthol added into CA dope and MOF controls continue to be submitted to CTSD on a weekly basis to observe aging effects on menthol delivery. Additional filters with menthol added into CA dope have been requested from Eastman to deliver a target 0.50 mg menthol in smoke. Cigarettes were evaluated by Flavor Technology personnel for subjectives.

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**PROJECT NUMBER :** 2305  
**PROJECT TITLE :** Internation Flavor Development and Brand Support  
**PROJECT LEADER :** J. Swain  
**PERIOD COVERED :** July, 1991

## **I. PROCESS SUPPORT**

- A. **Objective:** To develop and evaluate process modifications to maintain subjective and physical quality.
- B. **Status:** POL 0385 of Marlboro control and test cigarettes with reduced humectant RL's (46% solubles) and RCB has been released. Results are anticipated August 13, 1991.

Marlboro prototypes with 24% RCB from 330 ft./min. versus 350 ft./min. line speed trials at the BL Plant showed no obvious differences on the Flavor Technology panel. Further trials are being evaluated with RCB from 330 ft./min. vs. 350 ft./min. line speed with different dryer profiles prior to testing a higher line speed.

Marlboro cigarettes from the latest trial at the KC Spotswood plant for potential substitution of RLTC were made to qualify SRL-35. Subjective differences were shown with SRL-35 from the latest trial at the KC Spotswood plant at several substitution levels for RLTC. Discussions of potential modifications are in progress prior to a trial in September.

## **II. CAST LEAF PROGRAM**

- A. **Objective:** To develop and evaluate flavor systems for cast sheet utilization.
- B. **Status:** Additional flavor formulations substituted for PM1 flavor were subjectively evaluated in Cast Leaf Lab sheets (#143-165) with selected levels of other additives. From comparisons to a blend with 50/50 RL and RLB, Cast Leaf Lab formulations with increased isosweet levels tended to be harsher with more throat irritation. Individual and combinations of flavors which showed subjective attributes most similar to the control blend were selected for further evaluation replacing RLB.

Subjective evaluations are in progress on sheets made from combinations of Guar and DAP/ammonium hydroxide binder treatments to select formulations for tests in blends.

## **III. NET PROGRAM**

- A. **Objective:** Provide Flavor Technology Support for the New Expanded Tobacco Program.
- B. **Status:** The Flavor Technology Panel, Richmond Panel and MC Panel evaluated the Marlboro Ultra Lights with cased BLDET vs. cased NET BLDET and PM Lights

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with cased BFET compared to cased NET BFET. No subjective differences were noted.

PM Lights and Super Lights have been made to compare cased BFET to cased NET BFET. Subjective evaluation will be initiated as soon as analyses are completed.

#### IV. OPERATIONS SUPPORT

- A. **Objective:** To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.
- B. **Status:** POL's 0397 and 0398 results showed no differences for the Marlboro KS cigarettes with 100% reduction of alcohol in the BTC. Plans are in progress to make an initial trial with the 100% reduction of alcohol in the BTC of Marlboro at Louisville prior to extended trials. Evaluations are in progress for the six Marlboro models with alcohol reduction, humectant rearrangement and low humectant sheet by the MC panel.

The initial factory trial of 50% reduction of alcohol in the BTC was conducted at the Manufacturing Center. A storage study of the cigarettes has been initiated on the factory trial model. Merit KS with 100% ETOH reduction in the BTC evaluation has been completed by the MC panel. No differences were detected.

Semi-Works trials were completed for a Marlboro KS model with BTC-100% alcohol reduced, AC-20% alcohol reduced vs. Marlboro KS Control. Flavor Technology and Richmond Panel results showed no differences.

A Semi-Works run was completed for a Marlboro KS model with BTC-100% alcohol reduced, AC-30% alcohol reduced vs. Marlboro KS Control. Cigarettes have been submitted for analyses prior to internal panel evaluations.

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PROJECT NUMBER : 2307  
PROJECT TITLE : Basic Flavor Investigation  
PREPARED BY : R. W. Hale  
PERIOD COVERED : July, 1991

## **I. ANALYTICAL SUPPORT**

A. **Objective:** To provide analytical support for activities related to development and application of flavoring materials.

B. **Results:**

Two hundred twenty-six analysis requests were completed during the month of July, and one hundred seventy-three new sample requests for the month were received.

1. Samples for Project 2304: Flavor samples, purities and headspace analysis were completed during the month.
2. Samples for Project 2305: Casings and aftercuts for SRL qualification, Grain and storage study for flavors and casings. Marlboro Standardization Run IX analyses were completed during the month.
3. Samples for Project 2306: Marlboro concentrates, preblends, casings, MS/MSA POL, Marlboro Extra Lights POL were completed in July.
4. Samples for Project 5001: Residual solvents and GC/headspace analysis for packaging materials were completed during the month of July.
5. Headspace/GC analyses of Marlboro, Marlboro Lights (Egypt) and Barclay UL (Switzerland) were completed in the month of July.

## **II. FLAVOR INVESTIGATION**

A. **Objective:** To develop new basic and applied flavor technology in support of new product development objectives.

B. **Results:**

1. The efforts to identify the off-odor and taste in triacetin and glycerin are ongoing. Subjective evaluation of cigarettes prepared with good and bad glycerin is underway and results of the smoking panel should be ready by August 1, 1991.
2. A repeat of the sucrose inversion in Burley spray is underway to confirm the data from the previous runs.

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PROJECT NUMBER : 4016  
PROJECT TITLE : Paper Technology  
SECTION LEADER : S. D. Baldwin  
WRITTEN BY : G. H. Bokelman  
PERIOD COVERED : July, 1991

I. AMBROSIA (B. Geiszler and S. Tafur)

A. Objective: To develop cigarette wrappers for evaluation on low smoke Ambrosia models.

B. Results: Cigarettes were made with three Kimberly-Clark papers having initial basis weights of 45 g/m<sup>2</sup> and different permeabilities that had been sized with monopotassium phosphate. Sidestream reduction was measured on these cigarettes. Models made with paper at 2.3 Coresta base porosity showed 63% sidestream reduction compared to Marlboro Lights 100; models with 3.7 Coresta base paper gave 58% sidestream reduction; and models with 4.8 Coresta base paper gave 56% sidestream reduction.

The sizing of CR-2950 (an  $\alpha$ -hexylcinnamaldehyde release compound) on a 47.5 g/m<sup>2</sup>, low porosity calcium carbonate paper previously sized with 13% monobasic potassium phosphate was completed. Some of the cigarettes were placed in the jungle and desert rooms for four days and then submitted for HPLC analyses to determine the stability of the release compound on paper. Control cigarettes were kept in a conditioned lab (75°F, 60% RH).

C. Plans: Additional handmade cigarettes, having papers sized with CR-2950, will be analyzed for various smoke components and evaluated for subjectives.

II. MAGNESIUM CARBONATE PAPERS (G. Bokelman, S. Tafur, B. Goodman, and B. Floyd)

A. Objective: To develop a magnesium carbonate (magnesite) paper for a low sidestream product.

B. Results: Cigarettes in 24.0mm circumference configuration were made in the Semiworks with binary and ternary magnesite papers coated with different levels of potassium succinate. Some of these same papers were also used in making 23.0mm circumference models. The binary paper coated with 7% potassium succinate on 24.0mm circumference cigarettes gave 57% visibility reduction. The ternary paper with 6% potassium succinate on 23.0mm circumference cigarettes produced tar and puff counts equal to the control of the same construction made with conventional paper. This ternary paper model gave 70% sidestream reduction, compared to Marlboro Lights 100.

Cigarette models made with magnesite papers were evaluated for their degree of ash flaking. The following results were obtained: (1) ternary papers tended to have more

severe flaking than binary papers, (2) the more porous papers gave worse flaking than the tighter papers, (3) flaking was much worse with no additive on the paper, and (4) papers sized with potassium citrate were more flaky than those sized with potassium succinate.

A series of ternary magnesite handsheets at 45 g/m<sup>2</sup> basis weight with varying porosities were evaluated for sidestream reduction and staining. There was no loss in sidestream reduction going up to 10 Coresta. Going from 10 to 14 Coresta produced only a small fall off in sidestream reduction (60% to 55%). Above 14 Coresta there was a rapid fall off in sidestream reduction. These data suggest that a ternary magnesite paper at ~12 Coresta might be optimum with respect to both paper staining (which decreases with increasing porosity) and sidestream reduction.

Binary magnesite handsheets having 45 g/m<sup>2</sup> and 55 g/m<sup>2</sup> basis weights were compared at two different total filler contents (40% and 45%). Somewhat surprisingly, it was found that increasing the total filler content from 40% to 45% had virtually no effect on the properties of the handsheets. In particular, the increase in filler (and magnesite) content did not improve sidestream reduction.

As part of efforts designed to address ash appearance of magnesite papers, sizing requests were submitted for CMC and PVOH (polyvinyl alcohol) on selected magnesite papers to determine if these additives affect ash flaking or staining.

- C. **Plans:** An action plan has been developed by Philip Morris, Ecusta, and Midwest Custom Services, Inc. (MCS) to further minimize trace contamination with organic compounds during the next preparation of ground Baymag magnesite at MCS. This next preparation of ground magnesite will produce enough material to support multiple mill runs of magnesite paper at Ecusta.

### III. BLEND COMPOSITION (G. Bokelman and J. Stimler)

- A. **Objective:** To analyze the blend compositions of samples of expanded tobaccos isolated by CTSD from cigarette brands produced by RJR and B&W.
- B. **Results:** It appears that neither company is deliberately expanding Oriental leaf; only trace quantities of Oriental are present in their expanded products. It is probable that the starting material for the RJR expanded product is roughly a 5:3 ratio of bright lamina/burley lamina. It appears that the starting material for the B&W expanded product may be roughly a 2:1:1 ratio of burley lamina/bright lamina/stems.
- C. **Plans:** Blend composition analyses will continue to be performed as requested.

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**IV. SELECTIVE FILTRATION** (B. Geiszler, A. Finley)

- A. **Objective:** To identify smoke components whose deliveries correlate with the subjective performance of low sidestream cigarette models.
- B. **Results:** Neural network analysis techniques have been applied to the analytical and subjective smoking results for fourteen cigarette models. Good correlations were obtained between the chromatograms for the acidic fraction of smoke condensate and three subjective rating categories.
- C. **Plans:** Neural network analyses also will be performed on the basic and neutral fraction chromatograms for these same cigarette models.

**V. TIPPING PAPER PROGRAMS** (B. Geiszler)

- A. **Objective:** To develop improved tipping papers for current cigarette products.
- B. **Results:** A Bristol FF 100 model made in the factory with tipping printed with low silicate inks was evaluated by the Flavor Panel versus the production control cigarette. No differences were reported between the two cigarettes.

Marlboro Lights KS tipping printed with a modified lip release coating formulation on Ecusta 30136 basesheet had given a high level of coating ruboff during perforation. No ruboff problems were experienced for the same coating applied to Kimberly-Clark's GSR-236M2 basesheet. Ecusta's tests have shown a slightly slower absorption rate for their sheet relative to the KC sheet, and they plan to modify that property for evaluation.

- C. **Plans:** Machinability testing will be conducted for promising tipping candidates.

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PROJECT NUMBER : 4017  
PROJECT TITLE : Paper Making Processes  
PROJECT LEADER : R. M. Rogers  
PERIOD COVERED : July, 1991

## **I. LOW SIDESTREAM SMOKE AND LAB SUPPORT**

- A. **Objective:** Develop proprietary cigarette papers for low sidestream smoke.
- B. **Results:** Pilot paper runs are scheduled at Maine for two 35 g/m<sup>2</sup> cigarette papers, with either Omega Fine or Albacar carbonates. The Omega Fine carbonate was obtained from Pfizer as a 10% slurry rather than as a dry powder. Handsheet evaluations indicate that the Omega Fine reduces sheet porosity relative to Albacar carbonate necessitating a higher freeness flax stock to achieve 30 Coresta target. Formation may be a problem at this elevated freeness level.

The freeness/porosity relationship of the three Pfizer carbonates appears to be a function of filler particle size, the smaller the size the lower the porosity at equal freeness. This relationship was not as evident with previous samples of Omega Fine which were obtained as a dry powder. Apparently the slurry form of Omega Fine enhances the carbonate's dispersant characteristics.

- C. **Plans:** Continue supporting the production of pilot quantities of cigarette papers.

Develop the capability to produce 35 g/m<sup>2</sup> papers at Maine.

## **II. BANDED PAPERS (TOMORROW)**

- A. **Objective:** Incorporate cross directional bands of fiber and/or filler in cigarette papers in order to vary cigarette burn rate.
- B. **Results:** Porosity evaluations and chemical tracer analysis (propyl paraben) of banded papers produced at Beloit indicate that insufficient Cellulon was applied to the web. High speed video shots of the banded device indicate that not only was a portion of the Cellulon slurry rejected at the coater, but a portion also remained in the grooves after contact with the sheet. Both actions may have resulted in insufficient Cellulon being applied to the web. A program is being developed to modify slurry rheology and tack characteristics to improve application level.
- C. **Plans:** Develop a program to modify the printing characteristics of Cellulon for use by the rotogravure type unit.

### III. PROPRIETARY FILTER MATERIAL

- A. Objective: Develop a proprietary filter material in web form.
- B. Results: CA dispersion and softwood refining trials were conducted at James River's Gouverneur tissue mill this month. Refining results were similar to trials conducted at Neenah with the exception of fiber length. Since higher power levels were used to prepare the softwood, the fiber length was lower. Refining conditions can be modified to increase fiber length.

The dispersion characteristics of CA were evaluated in Gouverneur's new 20,000 gallon chest using low shear and low consistency in the presence of a chemical dispersant. CA flock balls were observed in the chest after only 30 minutes of operation. It is hypothesized that the dispersant was stripped from the CA during agitation which permitted the CA to mechanical entangle. Lab evaluations are in progress to determine if the addition of softwood will inhibit the formation of flock balls. If this approach is successfully, an additional CA dispersion trial will be conducted.

A number of web models were produced at James Rivers Neenah research facility this month. PM webs (75% cut CA and 25% refined softwood) with and without dispersant (2% CB-10) were completed. A 75/25 CA/Cellulon web was produced using homogenized Cellulon without crepe since the web failed to adhere to the Yankee. The inclusion of 10% softwood permitted inclusion of crepe (75/15/10 CA/Cellulon/softwood). An 80/20 CA/Fibrids sheet was also produced without crepe due to the web's failure to adhere to the Yankee. A 75/25 CA/cotton sheet was completed with crepe as a subjective option for CA/softwood.

- C. Plans: Identify a CA dispersion scenario that will permit production of developmental quantities of PM web at James River's Gouverneur tissue mill.



PROJECT NUMBER : 4018  
PROJECT TITLE : Paper Product Development  
PROJECT LEADER : B. L. Goodman  
PERIOD COVERED : July, 1991

I. REDUCED SIDESTREAM CIGARETTES (B. Goodman, B. Floyd)

A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream smoke.

B. Results:

Superslims Single Wrap: Sidestream visibility testing was performed on samples of regular and menthol Superslims produced in the initial production of single wrap cigarettes. Visibility reduction was comparable for the regular and menthol versions, ranging from 61% to 67%, compared to Marlboro Lights 100. Testing will continue on subsequent production samples.

Ambrosia: Cigarettes were made with these papers, which were designed to duplicate the Ambrosia POL's from last year. The sidestream reductions measured on the cigarettes were comparable to those from the POL tests, but mainstream data differed. Models made with paper at 2.3 CORESTA base porosity showed 63% reduction compared to Marlboro Lights 100; models with 3.7 CORESTA base paper gave 58% reduction; and models with 4.8 CORESTA base paper gave 56% sidestream reduction.

Paper samples from the different mill runs of 45 g/m<sup>2</sup> low sidestream paper from Kimberly-Clark were examined microscopically with no observed differences in flax and shive content.

Magic: Cigarettes in 24.0 mm circumference configuration were made in Semiworks with binary magnesite paper coated with three levels of potassium succinate, and with ternary paper with some of the same additive levels. Two of the papers were also used in making 23.0 mm circumference models. The binary paper coated with 8% potassium succinate on 24.0 mm circumference cigarettes gave 52% visibility reduction compared to Marlboro Lights 100. This model burned faster than the one with conventional paper, resulting in a mainstream tar delivery of 1mg less than the control. The ternary paper with 6% potassium succinate on 23.0 mm circumference cigarettes gave 70% sidestream reduction and resulted in tar and puff counts equal to the control of the same construction with conventional paper.

The 24.0 mm circumference models were evaluated for their degree of ash flaking during puffing. The ternary papers tended to have more severe flaking than the binary papers; flaking was much worse with no additive on the paper; the more porous papers resulted in more flaking than the tighter papers; and papers sized with potassium citrate were more flaky than those sized with potassium succinate.

- C. **Plans:** Continue the monitoring of sidestream visibility reduction for single wrap Superslims cigarettes.

Coat magnesite papers with additional types of additives and make cigarettes for evaluation of sidestream visibility and ash characteristics.

Perform coating and slitting services as needed of experimental papers for machine-made cigarettes.

Continue single port measurements on handmade cigarettes.

## II. PAPER SPECIFICATIONS

- A. **Objective:** To develop cigarette wrapper specifications for new product development and support of current products.
- B. **Results:** Bobbins of 10-058-A production 26.5 g/m<sup>2</sup> cigarette paper have been obtained at 38, 42, and 47 CORESTA from Cabarrus. In addition, Kimberly-Clark located and shipped a bobbin at 50 CORESTA, which was the highest porosity that could be found. Cigarettes will be made to determine the effect on delivery of porosities at the limits of the current porosity range.
- C. **Plans:** Determine the outer Coresta ranges that can be accepted on a product.

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**PROJECT NUMBER :** 4022  
**PROJECT TITLE :** Product Development, Affiliates and Licensees  
**PROJECT LEADER :** R. E. Tinker  
**PERIOD COVERED :** July, 1991

**I. L&M PROJECT (Venezuela)**

- A. **Objective:** Develop an acceptable L&M product for the Venezuelan market.
- B. **Status:** L&M prototypes (80mm) have been produced in Venezuela and submitted for C.I.'s in Richmond.
- C. **Plans:** If C.I.'s are within specifications, submit samples to the Richmond Panel for final approval.

**II. PROJECT ANDES (Panama)**

- A. **Objective:** Develop an acceptable L&M Menthol for Panama.
- B. **Status:** C.I.'s have been completed on 80mm FTB and samples submitted to Richmond Panel for subjective approval.
- C. **Plans:** If Richmond Panel approves subjectively. Panama will launch.

**III. MARLBORO IMPROVEMENT PROGRAM (Brazil)**

- A. **Objective:** Make blend changes to improve the subjective character of the Brazilian Marlboro family.
- B. **Status:** Brazil has produced three new prototypes (0% Oriental, 7.5% Oriental & 10% Oriental). Samples were submitted for C.I.'s on 7/13/91.
- C. **Plans:** When C.I.'s are completed, submit samples to Leaf and Richmond Panel for subjective evaluations.

**IV. L&M PROJECT (Mexico)**

- A. **Objective:** Develop an acceptable L&M product using USA produced casings/flavors.
- B. **Status:** C.I.'s are complete on three prototypes produced in Mexico.
- C. **Plans:** Discuss C.I. results and possible blend modifications with Richmond Leaf Department.

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**PROJECT NUMBER :** 4030  
**PROJECT TITLE :** Product Development, Export  
**SECTION LEADER :** J. N. Smith  
**WRITTEN BY :** R. S. Slagle  
**PERIOD COVERED :** July, 1991

**I. BLACK TOW ELIMINATION**

- A. **Objective:** Eliminate the existing black tow items 5.0x36,000, 3.4x44,000, 3.9x40,000 and replace them with existing white tow items.
- B. **Status:** Inventories of black tow item 5.0/36,000 were depleted on 7/31/91, and brands affected, including Parliament 100mm SP and FTB, were converted to 5.0x35,000 white. All Parliament products, domestic and export are now specified with white tow.
- C. **Plans:** Black tow items 3.9x40,000 and 3.4x44,000 are forecasted for depletion in October and December respectively.

**II. PARLIAMENT LIGHTS KS FTB (Korea)**

- A. **Objective:** Introduce a Parliament Lights KS FTB line extension into the Korean market to compete with 88 Lights and Mild Seven Lights.
- B. **Status:** PMI consumer test results were received 7/18/91, and the "Softer" version was selected for launch. Production of 41.55 million cigarettes began 7/23/91; for an 8/4/91 arrival date in Korea and a 8/12/91 launch. An additional order for 21 million cigarettes has also been received.
- C. **Plans:** A KS Soft Pack line extension of Parliament Lights in Korea is scheduled for August CPC submission.

**III. PROJECT BOLD (Europe)**

- A. **Objective:** Develop a 2.0mg ISO tar version of BOLD KS for consumer testing in Switzerland and Germany.
- B. **Status:** Two hundred thousand cigarettes of the 2.0mg ISO tar version of BOLD KS were produced in Semiworks 7/22/91. Cigarettes have been subjectively and analytically evaluated and released for shipment, scheduled for 8/8/91 to Philip Morris Europe R&D.

- C. Plans: BOLD will be consumer tested in Germany versus modified R1 and an improved PM Ultra prototype. Testing will also be conducted in Switzerland versus Barclay Ultra and the improved PM Ultra.

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**PROJECT NUMBER :** 2304/4031  
**PROJECT TITLE :** Product Development, U.S.A.  
**SECTION LEADER :** C. B. Altizer  
**WRITTEN BY :** G. N. Yatrakis and J. L. Spruill  
**PERIOD COVERED :** July, 1991

## **I. LOW TAR/HIGH FLAVOR**

### **PROJECT BOLD**

A. **Objective:** Develop 1mg 85mm and 2mg 100mm regular and menthol products competitive with Now and Carlton.

B. **Status:** A confirmation factory trial was completed at Cabarrus on July 26 which included a primary run as well as a cigarette manufacturing test of two tipping paper pressure drops for both 83mm and 100mm box designs. The trial product has been submitted to both QA and R&D for analysis.

Data has been received for the 85mm and 100mm 3mg and 4mg tar baseline matrix using blend, ventilation, and cigarette paper as the variables. Subjective evaluations have warranted a request to Filtrona for filters to achieve a lower RTD on both the 3mg and 4mg models. Primary/Make-Pack requests are being submitted for 85mm and 100mm models using two blends [298 (38% JET)/244 (50% JET)] and three filters (current DPCC/modified DPCC/1.8 DPF tow).

C. **Plans:** Continue development work to optimize subjectives and achieve target deliveries for 3mg and 4mg products. Finalize product specifications for BOLD 1mg KS/2mg 100mm.

## **II. PROJECT AMBROSIA**

### **AMBROSIA I**

A. **Objective:** Develop a 23.0 circumference aromatic sidestream product as well as a 24.8 circumference 85mm and a 24.0 circumference 100mm and apply this technology to other products.

B. **Status:** Two prototypes incorporating CR-2950, and their respective controls, have been received from Paper Technology. Subjective mainstream and sidestream evaluations are in progress.

Ambrosia I POL's 0591 (100mm Regular, 24.8mm, .15mg/cigt A-150) and 0676 (100mm menthol, 24.8mm, .15 A-150) were shipped on July 22 and July 29.

Ambrosia I (85mm/24.8mm regular and menthol) models were made in Semiworks on July 24. Samples included 9mg and 11mg models for subjective evaluation.

- C. Plans: Evaluate and select most appropriate 85mm prototype for consumer testing.

### III. PROJECT MARLBORO

- A. Objective: Design and develop Marlboro products as possible line extensions.
- B. Status: Product specifications (preliminary) are complete for MF Medium 100's SP and FTB and have been distributed to Operations Services. Plans call for Cabarrus to manufacture the FTB and the Manufacturing Center to make the soft pack. Currently, we are in the process of arranging factory trials at these locations to finalize tipping paper pressure drop and evaluate one row of perforation instead of two rows.
- C. Plans: Finalize factory trial schedule and produce/evaluate product.

### IV. BRAND EXTENSIONS

- A. Objective: Design and develop extensions and modifications to existing brand families.
- B. Status:
- Parliament Lights Menthol: Factory trials for the 100's SP, LS 20's FTB, and LS 10's FTB were completed at Stockton Street. Product has been submitted for analytical evaluations.
- B&H KS: Cigarette designs have been finalized for POL prototypes using two menthol production fillers and two production menthol-on-foil levels.
- C. Plans: Complete subjective evaluations of Parliament Lts menthol products. Fabricate and evaluate POL prototypes for B&H KS menthol.

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PROJECT NUMBER : 5001  
PROJECT TITLE : Packaging Studies  
PROJECT LEADER : B. Mait  
WRITTEN BY : C. Kroustalis  
PERIOD COVERED : July, 1991

## **I. PACKAGING STUDIES**

- A. **Objective:** Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.
- B. **Status:** Residual printing solvents were determined by headspace/GC on 22 samples of packaging materials including labels, inks, inserts, cartons and totes from domestic and Brazilian packaging materials. GC/MS analyses were also performed on some of these materials to identify unknown components.

The aging study for Project GOLD of samples manufactured at Cabarrus was completed. There were no significant differences in pack adhesion between standard and pre-applied adhesive softpack products. The first label case design failed under simulated warehouse storage (label blocking was observed). A second warehouse simulation storage study was initiated with a redesigned case.

The New Orleans warehouse stacking test for Project Hairy of long and short grain FTB samples in 10, 11 and 12 pt. board calipers will be completed the week of August 5, 1991. The 11 and 12 pt. boards passed subjectives. Subjectives for the 10 pt. board have not been completed.

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**PROJECT NUMBER :** 1309  
**PROJECT TITLE :** Cast Leaf Development  
**PROJECT LEADER :** G. Gellatly, G. D. Keritsis  
**PERIOD COVERED :** July, 1991

### **CAST LEAF DEVELOPMENT**

- A. Objective:** Develop subjectively and physically acceptable reconstituted tobacco sheets for domestic and international application using Cast Leaf technology and proprietary binder systems.
- B. Results:** **Cast Leaf Pilot Plant** - The State approved the air permit for pilot plant construction. Installation of structural steel, decking and electrical conduit was begun. The sheet dryer and coater will be delivered in mid-August, as well as other major pieces of equipment.

**Cast Leaf Development** - The physical quality of guar gum sheet product using guar in the binder and guar blended in the tobacco was not found to be significantly different. The addition of guar in tobacco will simplify a commercial operation because guar gum binder can become very viscous in a holding tank. It will also improve product uniformity by maintaining a fixed guar gum hydration time. Quantification of the effect on smoke delivery of guar gum products of different sheet weights and different particle sizes of feedstock was delayed by the Semiworks small scale facility shut down.

A method of making a "hybrid" sheet was developed in the laboratory. This sheet is a combination of guar gum and DAP/ammonia binder to achieve the intermediate subjective characteristics of RL and BL. There was a problem making this sheet because of the interference of ammonia/DAP with guar gum hydration. However, when the ammonia is added after guar gum hydration, this problem is overcome.

It was shown that reduced tobacco particle size and the addition of woodpulp improved guar gum sheet quality over a range of moisture contents (14-21% OV).

Several other binders (Locust bean, Xanthan, and Acacia) were used for sheet making for evaluation of their subjective character.

**Cadiz** - Initial trials with the slurry deaeration equipment showed a marked improvement in sheet quality when the air was reduced in the slurry. Pinholes in the sheet were visibly reduced and the tensile strength of the sheet was increased over 50%. A test plan was formulated for Cadiz to optimize deaeration conditions before PM trials are run in August.

**BL Plant Capacity Increase** - RCB products at higher plant throughput have not been subjectively identical to control RCB. The sheet dryer temperature profile appears to be a major factor affecting subjective character. Higher slurry solids using finely ground tobacco would maintain the drying profile at increased line speed and such a trial is anticipated in September.

**Alternate Sheet Suppliers** - RLTC made in Schweitzer (Spotswood, New Jersey) was found to be subjectively different from PM-RLTC. The reasons for this difference were

discussed with Schweitzer. Plant modifications are being made prior to the next plant trial in September.

Cigarettes containing RLTC from the last American Tobacco Company trial are presently being evaluated subjectively.

RLTC from FTR arrived in the U.S.A. and will be evaluated as soon as possible.

**C. Plans:**

1. Continue binder and flavor development work to optimize the cast sheet formulation for USA and TSA.
2. Develop "hybrid" sheet technology on a larger scale.
3. Determine the effect of Cast Leaf process variables and formulation on cigarette smoke delivery.
4. Continue work with PM Engineering on the installation of the Cast Leaf Pilot Plant.
5. Run ASTA and SIVA trials in Cadiz in August 1991 to evaluate the effect of slurry deaeration, sheet weight, and flavor formulations on sheet quality and subjectives.
6. Rerun temperature and chemical profiles in packing cases in Cadiz during August climatic conditions to determine packout conditions to prevent ASTA product darkening.
7. Determine the conditions of OV, packout temperature, and packing density necessary to induce ASTA product darkening in the laboratory.
8. Determine the potential BL Plant capacity increase using more finely ground feedstock at increased slurry concentration and line speed.
9. Subjectively compare RLTC products from Schweitzer, LTR, and American tobacco with Park 500 RLTC for potential substitution due to increased demand.

PROJECT NUMBER : 1503  
PROJECT TITLE : Modified Smoking Materials  
PROJECT LEADER : W. A. Nichols  
PERIOD COVERED : July, 1991

## **I. STEMMERY WASTE**

- A. **Objective:** Determine the feasibility of reclaiming tobacco from stemmery waste.
- B. **Results:** A final report summarizing the results of this project was completed. Results were presented to the Tobacco Utilization Committee on July 11, 1991.
- C. **Plans:** No further work is planned on this program.

## **II. MENTHOL APPLICATION**

- A. **Objective:** Develop a method for applying encapsulated or thermal release menthol compounds.
- B. **Results:** A hotmelt applicator was used to apply a 10 mm wide coating of Polymic to cigarette paper. The melt point of the Polymic was modified by the addition of 10% Triacetin. Cigarettes were successfully produced and submitted for analysis. While satisfactory menthol delivery (.73 mg/cigt.) was achieved, the coating stains the paper behind the coal during smoking. Double wrapped cigarettes exhibited some staining as well.

After reviewing the cost of many of the synthesized method compounds, a decision was made to focus on application techniques for alginate/menthol capsules.

Various means for applying powdered materials to filler are being investigated. As most methods would involve using some carrier fluid, the stability of alginate/method capsules to drying is under evaluation.

- C. **Plans:** Alginate/menthol capsule stability testing will be completed. A small scale spray cylinder will be installed for coating experimentation.

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**PROJECT NUMBER :** 1806  
**PROJECT TITLE :** New Tobacco Processes  
**PROJECT LEADER :** T. C. Holland  
**PERIOD COVERED :** July, 1991

## **I. PRIMARY IMPROVEMENT PROGRAM**

- A. Objective:** Identify process and equipment modifications to provide a highly flexible primary operation capable of producing filler with significantly improved filling power at a lower operating cost. Characterize the existing primary process to establish baseline thermal history, chemical changes, and flavor reactions.
- B. Status:** Efforts are underway to evaluate the concepts associated with the New Primary Process. Testing has been initiated to measure the physical gains and subjective impacts of processing individual tobacco components (bright, burley, oriental, reconstituted) separately through the flavoring, cutting, and drying operations.

**New Process/Product Development** - Cigarette models are being made from filler containing Bright components that were processed independently and added back to a filler blend. The Bright component was processed under various scenarios to obtain an initial measure of the impact of cut width, Hauni tunnel treatment, and method of casing on the subjective and physical responses.

**New Process Modelling** - Witness software has been chosen for modelling the operational aspects of the Primary process. AT&T has been requested to submit a proposal to develop a software simulation of the existing Primary and the New Primary processes.

**Thermal Treatment** - Testing to establish the subjective and chemical responses to thermal treatment in the Burley dryer is in progress. The initial testing involving a range of drying conditions has been completed, including the filler analyticals, smoke analyticals, and subjective measures. Correlation studies will be completed in August.

**Steam Tunnel Evaluation** - The Sagemuller steam tunnel has been installed in the Semiworks prior to the Burley spray cylinder. Testing to demonstrate casing uniformity gains are in progress. Cigarettes have been submitted for subjective qualification. The project is on schedule to generate a recommendation by October 1991 to support an implementation decision at Cabarrus for incorporating steam tunnels prior to casing cylinders.

**Cut Filler Delivery Program** - Installation of the Rothmans Flow Thru Hopper in D Pilot Plant is 80% complete. The unit will be checked out and evaluated in a stand alone operation prior to installation on a Semiworks maker for product evaluation.

- C. Plans: Testing to determine the potential physical gains and subjective impacts associated with separate component processing will continue. The burley, oriental, and reconstituted components will be evaluated along the same procedure as for Bright. Blending trials are scheduled for August to establish the protocol required for blending individual cut filler components. Hauni tunnel trials to establish the optimum process conditions for each individual component are also planned to commence in August.

The schedule for developing a model of both the existing and New Primary process will be established in August.

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PROJECT NUMBER : 1810  
PROJECT TITLE : ART Process Development  
PROJECT LEADER : D. R. Fox  
PERIOD COVERED : July, 1991

## **I. LIQUID ABSORBENT TREATMENT PROCESS DEVELOPMENT**

- A. **Objective:** To develop separation, utilization, and disposal processes for the liquid absorbent effluent from the second-generation ART extraction process.
- B. **Results:** Final arrangements have been made for the production of nicotine citrate solution by an outside laboratory. This solution will be used as feedstock for the pilot activated sludge system. The first shipment of nicotine citrate is expected in late August, with the pilot system startup to follow shortly thereafter.

For the pilot system, primary effluent from Waste Treatment will be used to dilute the nicotine citrate. Deionized water has been used as the dilutant for the bench-scale system but it had to be supplemented with ammonium bicarbonate to increase the nitrogen content of the feed stream to support microbial growth. The primary effluent is expected to provide enough supplemental nitrogen so that AB addition will not be necessary.

The bench-scale system continues to operate with no detectable nicotine in the effluent and COD's around 100 mg/l. Analysis of the waste sludge has also shown no detectable nicotine. The only operational problem encountered has been a poorer settling sludge which is being caused by the nitrogen deficiency as the AB addition was decreased in an attempt to reduce or remove it from the feedstock.

C. **Plans:**

1. Start up pilot scale treatment system in the Bermuda Hundred Pilot Plant.
2. Use Park 500 primary effluent as dilutant for pilot scale feedstock.

## **II. BL PLANT WATER TREATMENT**

- A. **Objective:** Identify and develop techniques for reducing levels of targeted constituents (e.g., nitrates and phosphates) in the discharge water from the BL Plant.
- B. **Results:** The batch tests of a modified burley stem washing process have been completed. The results were generally negative from an environmental point of view. The ratios of phosphorous and BOD to nitrate in the extract was not significantly different from the current process. For a given nitrate extraction target, varying the time and temperature of the wash will not significantly reduce levels of the other pollutants.

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The tests did suggest that a more rapid wash, possibly using smaller quantities of water, may still achieve the nitrate reduction in the stems. Such testing will require a continuous extraction system.

Preliminary flocculation studies have been conducted with ferric chloride, alum, and several polymeric flocculating agents. Results have shown very good removal of total suspended solids with more moderate removal of BOD, phosphorous, and alkaloids. The treatments have had little effect on nitrate. The BOD and phosphorous removals have been less than anticipated, so we are investigating the experimental methods and consulting with McNamee and polymer manufacturers on the problem.

**C. Plans:**

1. Complete flocculation evaluation.
2. Complete conceptual evaluation of SBR and issue purchase requisition.

**III. BL PLANT AIR TREATMENT**

- A. Objective:** Identify and develop techniques for reducing levels of targetted constituents (e.g., ammonia and nicotine) in the discharge air from the BL Plant.
- B. Results:** Work has begun on three aspects of the BL air scrubbing project. We are working with Engineering on developing a tall stacks/scrubbing scenario for atmospheric dispersion modelling. When completed, this scenario will be turned over to Black & Veatch for the modelling work. A test program is being developed with Chemical Research to develop equilibrium data for the nicotine/water system to aid in designing and evaluating the scrubber. Resources are also being identified to assist in developing a design for a pilot scrubbing system.
- C. Plans:** Continue to develop aspects of the tall stacks/scrubbing scenario, leading to pilot testing of the concept.

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**PROJECT NUMBER :** 1812  
**PROJECT TITLE :** New Expanded Tobacco  
**PROJECT LEADER :** E. B. Fischer  
**PERIOD COVERED :** July, 1991

## **I. BATCH GASEOUS CO<sub>2</sub> IMPREGNATION**

**A. Objective:** Define process parameters for a batch gaseous CO<sub>2</sub> impregnation process.

**B. Results:** Improvements to the gaseous CO<sub>2</sub> impregnation cycle at the Bermuda Hundred facility were successfully implemented resulting in further reductions in the total cycle time. The CO<sub>2</sub> flow rate during the cooling step was successfully increased to 1000 lbs/min without adverse affects on the bed temperature uniformity or bed pressure drop. At this flow rate, target bed temperatures were achieved within 6 to 7 minutes resulting in a total time under pressure of approximately 22 minutes. The depressurization step took 10 of the total 22 minutes and was rate limited by the existing recovery compressor in Module 3. With the properly sized equipment for this recovery step and a loading and unloading duration of approximately 15 minutes, a total cycle time of approximately 33 minutes to meet the targeted 5000 lbs of expanded tobacco per hour is achievable.

Gaseous CO<sub>2</sub> impregnation trials at 950 psig were completed to determine if this operating path will successfully yield a shorter cycle time. For these trials, the tobacco bed was cooled to a temperature of approximately 20 °F by flowing CO<sub>2</sub> gas through the bed at 400 psig. The target post-vent bed temperature of approximately 0 °F was not achieved using this operating path. Final post-vent bed temperatures ranged from +10 to +20 °F which resulted in a relatively unstable impregnated filler.

Testing was conducted to evaluate the effect of primary processing parameters including cutting OV, cutting temperature, cut width, and sugar casing on filler size feeding the NET process. Results indicated that there were no major differences in sieve size for any of the variables except cutting OV where a cutting OV of 24-25% was best. In addition, testing was completed to compare the survivability of 25 vs. 30 cpi through the NET process. The filler with 25 cpi consistently showed improved survivability over 30 cpi.

**C. Plans:** A series of 5 consecutive runs with 15% OV tobacco and 800 psig impregnation pressure is planned using the current operating cycle to establish the effects of continuous operations on temperature profiles and impregnation.



## II. CONTINUOUS IMPREGNATION PROCESSES

- A. **Objective:** Develop a continuous impregnation process to improve the subjectives of expanded tobacco while maintaining equivalent cigarette filling power to the existing process.
- B. **Results:** Work is continuing on the design and construction of a short cycle impregnation (SCI) pilot plant. The tobacco feed system for the SCI process has been defined and will consist of Rothmans' TWD feeders. Metering and tobacco degradation tests were successfully conducted at Rothmans in Canada. Based on the success of these tests two feed hoppers, two TWD feeders and controls have been selected for the pilot system. Bench scale tests were conducted to simulate tobacco transfer from the Rothmans' TWD to the impregnator. The results were used to establish charge tube and slide gate geometries for the feed system. A slide gate prototype was ordered to test the feeding of tobacco into the impregnator in less than three seconds.

The SCI pilot plant layout drawing and P&ID have been finalized. A critical path analysis of the project schedule is being conducted to improve the estimated start-up date of October 1991. Long delivery items have been identified and purchase orders have been initiated to expedite receipt of the equipment.

- C. **Plans:** Finalize the sequence of operation and track the project progress against scheduled milestones.

## III. EXPANSION AND REORDERING PROCESSES

- A. **Objective:** Define alternate means of puffing, drying, setting, and reordering impregnated tobacco to improve product subjectives and physical characteristics relative to the present DIET process.
- B. **Results:** The manufacturer of the spiral humid air reordering unit provided a new conveyor belt design for evaluation. Test results showed that the new design significantly improved tobacco handling. Work is continuing to optimize the performance of the reordering unit. Processing modifications to date have allowed expanded tobacco to be reordered from 3% OV exit the tower to 11.5% OV. Laboratory tests in environmental chambers have yielded results which indicate that with counter current flow a reasonable range of temperature and air velocities may be used to reorder tobacco without suffering a CV loss.
- C. **Plans:** Continue testing to establish optimum operating conditions for humid air reordering in the spiral unit. Relate expanded tobacco product attributes to tower operating variables through 8" tower data analysis.

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#### IV. CHEMICAL STIFFENING

- A. **Objective:** Define a process to chemically stiffen expanded tobacco which will reduce thermal treatment and the associated subjective degradation while maintaining cigarette filling power equivalent to the current process.
- B. **Results:** As a means to reduce the concentration of the acetate anion on the tobacco, a calcium acetate/bicarbonate mixed salt was evaluated on tobacco expanded in the 8" DIET pilot plant. It produced expanded tobacco with a 1.6 cc/g CV improvement over the control. These results are similar to those from samples treated with calcium acetate or calcium propionate. Based on these results a subjective evaluation of these additives is being planned to determine which additives show promise for further development and optimization.
- C. **Plans:** Determine the acid removal profiles for the additives. Evaluate the effect of pH adjustment of the additive solutions on CV results and acid removal profiles. Prepare samples for subjective evaluation including reduced acid products.

#### V. PRODUCT DEVELOPMENT AND EVALUATION

- A. **Objective:** To optimize both the physical and subjective characteristics of the NET process and to evaluate the inclusion of the various NET product options into present and future brands.
- B. **Results:** Marlboro Ultra Lights and PM Lights were made for comparative testing using ET blends that were DIET processed and NET processed for parity with DIET. The cigarettes were evaluated internally by Flavor Development and determined to have subjective parity. Additional testing by the MC panel is being scheduled.

In support of Project Tomorrow, sugar cased bright #10, C34 burley, and MT were made into a variety of expanded products using the NET process. The products, which were supplied to Product Development for cigarette evaluations, ranged from those processed with low thermal treatment (best subjectives) to those processed for maximum filling power (2-3% tower exit OV).

Cigarette models containing Marlboro blends of 12%, 22%, and 32% expanded tobacco were evaluated with low thermal treatment (tower exit OV 6-7%) NET, Manufacturing DIET, Pilot Plant DIET, and unexpanded Bright #10. Compacimeter results indicated that models containing 12% DIET and 22% NET had equal tobacco weights at a constant 3.0 mm firmness. In addition, cigarettes containing 32% NET maintained the same puff count, static burn time, and nicotine delivery as Marlboro cigarettes with 12% MC DIET.

- C. **Plans:** Continue testing to evaluate and demonstrate the benefits of NET processed tobacco.

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**PROJECT NUMBER :** 0008  
**PROJECT TITLE :** Computer Applications Division  
**WRITTEN BY :** J. Palesis, J. Blankinship, M. Allred  
**PERIOD COVERED :** July, 1991

## **I. Expert Systems Development**

- A. Objective:** Develop an Expert System for Cigarette Design.
- B. Results:** We have continued to incorporate into CigDES.1 the filter design features of version 8.1 of the Fortran cigarette model. All filter design functionality of the control cigarette design has been implemented. A few features of the prototype filter design still remain to be incorporated into KEE. In response to user request, we have also implemented a set of cigarette performance analysis functions which allow the user to explore how cigarette performance characteristics relate to cigarette design parameters. These features are based on the goal-directed search feature of CigDES which maintains a trace of the explored cigarette designs and their corresponding performance evaluation. After a goal-directed search is conducted, the user can command CigDES to graph, show, save, or print a mapping of the values of a specified design parameter against one or all other design or performance parameters.
- C. Conclusions:** The implementation of the correlation functions illustrates the power of KEE to deal with knowledge-based domains such as cigarette design. The high level implementation features of KEE made it possible to implement the new cigarette performance analysis functions in a matter of days. Once the basic interface between CigDES and the Fortran model is complete, these high level programming features of KEE will allow us to respond to user needs quickly to provide new functionality to both facilitate and enhance the cigarette design process.
- D. Plans:** After the new features have been reviewed, we will work with the primary user to test and evaluate the new version of CigDES.
- E. References:** Palesis, J.A., Dwyer, R.W., Leister, D.L., and Kao, J.W., "Transforming Mathematical Product Evaluation Models Into Expert Systems for Product Design," Proceedings of the 3rd International Conference on Industrial & Engineering Applications of Artificial Intelligence and Expert Systems, pp. 404-415, 1990.

## **II. Neurocomputing**

- A. Objective:** Develop programming techniques for use of the HNC ANZA Plus Neurocomputer and Neurosoftware. Investigate potential applications of neurocomputing to R&D problems, and apply where appropriate.
- B. Results:** The training methodology for Learning Vector Quantization was improved in several respects - principally the way in which the "repulsion" and "distance bias"

terms are controlled during learning. This avoids erroneous and inefficient weight adjustments early in training, and allows the network to focus on the decision boundaries rather than on equiprobability in the latter phase of training. As a result, classification performance is somewhat improved and training time is significantly reduced. Also, an "LVQ Test" program was developed to automate the manual process of performing n-fold cross-validations for various Kohonen layer sizes per class.

The analysis of data from the Selective Filtration Study using Learning Vector Quantization and pattern classification techniques is nearly complete. Neural networks were developed to classify chromatograms of model cigarettes according to their wrapper type, filter type, and overall liking.

- C. **Conclusions:** Learning Vector Quantization and other pattern classification techniques can be successfully used to help understand the relationship between cigarette design, cigarette analyticals, cigarette chemistry (e.g., chromatography), and subjectives.
- D. **Plans:** Complete pattern classification analysis for Selective Filtration Study, and issue a report.
- E. **References:** Research report 91-025, A Neural Network Model of Cigarette Liking.

### III. CTSD CIGARETTE LENGTH INSTRUMENT

- A. **Objective:** Develop software to acquire data from the Honeywell HVS 256 Edge/Width Gauge. This instrument will replace the current cigarette length instrument and data acquisition system.
- B. **Results:** A new base for the Honeywell HVS 256 was received from Development Engineering and the data acquisition software was tested. The instrument can now measure cigarettes from 67mm to 120mm. It has been installed in the CTSD Physical Testing lab and personnel have been trained. Information from the data acquisition system is updating the CTSD LAS2 database. This project is complete.
- C. **Plans:** Provide support as needed.
- D. **References:** CAD Project Request #4226

### IV. CTSD GAS PHASE/FTIR FIVE-PORT SMOKERS

- A. **Objective:** Develop a new data acquisition system (DAS) for the five-port smokers in the Cigarette Testing Services Division (CTSD) gas phase laboratory.
- B. **Results:** Two CTSD Gas Phase/FTIR Five-port Smokers using Laser Precision FTIR instrumentation are fully operational. This includes barcode support for sample identification, loading of results into the CTSD LAS2 database and training of lab

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personnel. Also, communications with the Eagle smoker controller were refined as were the peak detection algorithms.

C. Plans: Install a third and final unit in late August.

D. References: CAD Project Request #3979

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**PROJECT NUMBER :** 1101  
**PROJECT TITLE :** Entomological Research  
**PROJECT LEADER :** D. L. Faustini  
**PERIOD COVERED :** July, 1991

## **I. CIGARETTE BEETLE (CB) CONTROL PROGRAM**

- A. Objectives:** (1) To determine if phosphine resistance is occurring in the feral CB, (2) to determine if physiological differences are present in laboratory and field colonies regarding methoprene, and (3) to investigate alternatives to conventional tobacco pesticides.
- B. Results:** (1) Cultures maintained in methoprene were established using KABAT® feral CBs collected from Maury St. (Whrs. #28) and placed in a mobile insectary; (2) a proposal has been written to investigate esterase activity as an indicator of resistance to methoprene in the CB; and (3) to replace the inbred laboratory colony of CBs, wild populations are being collected and established in the insectary.
- C. Plans:** (1) Continue to collect KABAT® CB ferals and increase the culture size for use in CB physiological feeding studies; (2) set up a laboratory to perform esterase activity measurements; (3) wild populations of CBs will be collected from Kenbridge, Va. and other suitable sites for consideration as replacement cultures for the inbred lab colony; (4) continue to culture *Manduca sexta* larvae and collect purge fluid; and (5) S-hydroprene and kinoprene (IGRs) will be screened against the CB in feeding bioassays.
- D. References:**
- Minor, M. F. Notebook No. 9024, pp. 12 and 28.

## **II. SERVICE TO OTHERS**

- A. Objective:** Provide technical services to areas outside R&D.
- B. Results:** DataTrace® temperature profiles and CB efficacy studies in the Direct Cylinder Conditioner (DCC) were conducted in the Cabarrus M/C for PM Engineering personnel. CB efficacy tests in the Admoist Cylinder were conducted at Park 500 for PM Engineering personnel. Fourteen insect-related consumer complaints were handled for the QA Department.
- C. Plans:** Conduct CB mortality tests in the Direct Cylinder Conditioner start-up in Cabarrus (July 22-26, 1991).

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**D. References:**

Minor, M. F. Notebook No. 9024, p. 28.

Tenhet, S. W. Notebook No. 9091, p. 13.

**2022143179**

PROJECT NUMBER : 1720  
PROJECT TITLE : Analytical Microscopy  
PROJECT LEADER : V. L. Baliga  
PERIOD COVERED : July, 1991

**I. REDUCED SIDESTREAM/FILTRATION/PAPER TECHNOLOGY** (Baliga, Miser)

- A. **Objective:** Examine the ultrastructure of selected cigarette papers, paper additives, and filtration materials in support of the paper technology project.
- B. **Results:** Three filler materials were examined, two of which were sol-gels. The  $\text{CaCO}_3$  sol-gel, 7590-178, did not exhibit a rhombohedral crystal habit. Instead, it consisted of small equant-shaped particles  $\sim 0.1$  to  $10\mu\text{m}$  in diameter.<sup>1</sup> The silica sol-gel, code # 9087-4II, that had been applied to a cigarette wrapper paper surface, was relatively smooth but contained cracks and holes in the sol-gel surface.<sup>2</sup> A calcite material, code #9040-106, that was prepared at room temperature was examined and found to contain two particles types. One was a rhombohedra calcite,  $10\mu\text{m}$  or less along the long axis, and the second type was a cluster of equant particles arranged into spheres and ovoid shells.<sup>1</sup>

C. **References:**

1. Baliga, V., PM Notebook #8911, pp. 53-54.
2. Miser, D., PM Notebook #8975, p. 93.

**II. SUPPORT TO OPERATIONS** (Baliga, Miser)

- A. **Objective:** To provide support for operations.
- B. **Results:**

Foil overwrap, tipping paper, plug wrap, and cigarette wrapper paper from the new Winston packaging were examined. The foil overwrap was  $\sim 20\text{-}25\mu\text{m}$  thick and consisted of an outer print layer  $<1\mu\text{m}$  thick that was printed on a 2-3mm thick Al layer. This layer was attached to a polymer layer  $\sim 18\text{-}20\mu\text{m}$  thick which was covered by a final polymer layer that was  $\sim 2\mu\text{m}$  thick, dimpled, and full of holes. The Winston overwrap was about half as thick as the previously examined Premier overwrap.<sup>1</sup> The Winston overwrap contained only one,  $20\mu\text{m}$  thick polymer coat while the Premier overwrap contained 2,  $20\mu\text{m}$  thick polymer coats, one on each side of the Al layer.<sup>1</sup> Further examinations on an additional Winston 100s Lights sample showed that a thin layer of polymer covered the print on the outer surface. This layer was missed in the initial examination.<sup>2</sup> Additional analyses will be carried out on this sample.

Elements found in the print of the Winston overwrap were Ti in the white print, Ca, Cl, S, and Si in the red print, a small amount of Si and Cl in the gold print, and no



detectable elements in the creamy yellow or yellow print. Further examinations of this material will be done to determine if the extra polymer layer blocked the x-ray signal.

Plug wrap papers were similar from the new foil-wrapped Winston cigarettes and conventionally wrapped Winston cigarettes. Both contained wood pulp fibers. The same was true for the cigarette wrapper papers. Both contained the  $\text{CaCO}_3$  species, calcite and a small amount of aragonite, as well as wood pulp fibers. The tipping paper, however, from the foil wrapped Winston did not contain a lip release compound compared to the cigarettes from the conventional Winston pack.<sup>2</sup>

Two Spanish ASTA RCB samples were examined, one of which was processed with a versator before sheet making. The versator treated sample appeared to be slightly thicker in cross section and more dense, with fewer surface holes on the top surface. Both samples exhibited K- and S-containing crystals on the top and bottom surfaces. The versator treated sample contained larger patches of crystals versus the more widely distributed, smaller patches of crystals on the untreated sample.<sup>3</sup>

Tobacco particles and dust were examined and it was found that sand and hematite adhered tightly to the tobacco particles. These made sieving to remove the 'dirt' ineffective.<sup>4</sup>

#### C. References:

1. Baliga, V., "SEM Characterization of Premier Outer Wrap," Memo to P. Grantham, June 7, 1991.
2. Baliga, V., "The Morphological and Elemental Characterization of Winston Foil Overwrap," Memo to R. Fenner, July 11, 1991.
3. Baliga, V., PM Notebook #8911, p. 53.
4. Miser, D., PM Notebook #8975, p. 92.

2022143181

PROJECT NUMBER : 1752  
PROJECT TITLE : Molecular Structure Determination and Materials Evaluation  
PROJECT LEADER : G. Vilcins  
PERIOD COVERED : July, 1991

## **I. NMR ANALYSIS OF TOW ANCHOR ADHESIVES**

- A. **Objective:** To characterize the tow anchor adhesives by NMR procedures as part of the adhesive specifications program.
- B. **Results:** Six adhesives used as tow anchor adhesives (code: AD-010 - AD-015) were analyzed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR. The NMR results indicated that these types of adhesives were based on the polyvinyl acetate, (PVA), polymer system. With the exception of AD-011, the other five were PVA homopolymer. AD-011 was formulated with the poly ethylene vinylacetate, (PEVA), co-polymer. Polyvinyl alcohol, (PVOH), was found in all six samples. Three of the six contained a Benzoflex compound as a plasticizer.
- C. **Conclusions:** From the NMR results we were able to characterize the base polymer, calculate the ratio of PVA/PVOH, and identify the plasticizer in these tow anchor adhesives as a Benzoflex compound. The main difference between the tow anchor adhesives and tipping adhesives evaluated earlier was the amount of PVOH. Some of these adhesives contained PVOH at levels 5-10 times what was usually seen in the tipping adhesives. Although PVOH is widely used in the polymerization of polyvinyl acetate emulsions, the levels observed in the tow adhesives are probably due to PVOH added during formulation. It is well known that PVOH has many functions in an adhesive emulsion, from increasing the viscosity to improving adhesion.
- D. **Plans:** To continue to provide NMR support as needed.
- E. **Reference:**
- Bassfield, R., "NMR Analysis of Tow Anchor Adhesives," Memo to M. Zimmermann, July 16, 1991.

## **II. DETERMINATION OF NICOTINE IN NET AND DIET PROCESS CO**

- A. **Objective:** To compare the levels of nicotine in liquid carbon dioxide used in the DIET expansion process and in gaseous carbon dioxide used in the NET expansion process.
- B. **Results:** Carbon dioxide samples were collected from the MC DIET impregnators and from the D-pilot plant NET process using 500-ml high pressure sample bombs. The diet process  $\text{CO}_2$  was collected as a liquid at 450 psi into 16 bombs during eight fill/dump cycles of the DIET process. These samples were analyzed for nicotine using a GC equipped with a FID detector. The NET process  $\text{CO}_2$  was collected as a

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gas at 430 psi and 800 psi. Eight CO<sub>2</sub> samples were taken from one run at 430 psi and eight more at a second 800-psi run. The NET samples initially were analyzed by GC-FID, but the concentrations were below the limits of detection for the flame ionization detector. The samples were reanalyzed by GC with a NPD detector, which improved the lower detection limit for nicotine.

- C. **Conclusions:** NET process carbon dioxide had an order of magnitude less nicotine concentration than DIET process carbon dioxide. There was only a moderate increase in nicotine in the gaseous CO<sub>2</sub> when the pressure was increased from 430 to 800 psi.
- D. **Plans:** Continue to provide analytical support to the NET project.
- E. **References:**
  - 1. Thomas, E., PM Notebook 9082, pp. 76-83
  - 2. Pierotti, J., PM Notebook 8965, pp. 97-100

### III. INFRARED ANALYSIS OF R. J. REYNOLDS NEW OVERWRAPS

- A. **Objective:** To identify the chemical composition of the foil new pack overwraps for Winston Lights 100's and Winston Ultra Lights K.S. brands.
- B. **Results:** The old Winston Lights 100's had an overwrap of uncoated polypropylene. The tear tape also was polypropylene. The new Winston Lights 100's overwrap was composed of several layers of polymers, primarily polyethylene terephthalate. The tear tape was polypropylene. The coating on the printed foil side was a mixture of nitrocellulose and other components. The inside silver foil of the wrap had a layer of polyethylene terephthalate. The new Winston Ultra Lights K.S. overwrap was basically the same as the 100's. The tear tape was different and identified as polyethylene terephthalate.
- C. **Plans:** These are preliminary results and the investigation will continue.
- D. **Reference:**
  - Griff, M., PM Notebook #9085, p. 85.

2022143183

PROJECT NUMBER : 1757  
PROJECT TITLE : Analytical Flavor Specifications  
PROJECT LEADER : M. L. Zimmermann  
PERIOD COVERED : July, 1991

### FLAVOR AND ADHESIVES SPECIFICATIONS AND CERTIFICATION

A. Objective: To develop analytical and sensory specifications for current, incoming flavor materials and to transmit specifications and methods for monitoring specifications to appropriate groups. To certify that PMI export flavor materials meet GFO, to issue a certification of analysis and to transfer methodology and certification to the Flavor Center. To develop specifications and analytical methods for adhesives used by P.M. USA.

B. Results:

Requests to adhesive suppliers have resulted in the receipt of numerous additives that are commonly used in adhesive formulations, however, only three of the vendors have responded. A single vendor has been extremely helpful supplying over 21 individual ingredients. Two adhesive vendors were visited this month as a follow up to this request and some useful analytical techniques were observed. The examination of these materials has revealed various chemicals in a variety of compounded mixtures. Identification by literature searches and mass spectrometry is currently being done on the 52 materials being examined.

A sample of adhesive submitted for tracking was found to contain a particular component not currently acceptable for use in tipping adhesives. Contact with the vendor indicated a different component than was identified by mass spectrometry and profiling of reference compounds.

All of the tipping adhesive used in production at each of manufacturing locations in the states were analyzed by pyrolysis-GC/MS. A memo was issued.

A compilation of essential oils analyzed in ARD was issued this month.

Two methods for the determination of marker compounds are being written. Additional botanical materials have been obtained and secondary markers are being confirmed following location, processing and analysis of these materials.

Samples of specific fractions of vendor samples are being analyzed to identify those materials responsible for contributing to the contaminant present.

Following detailed technical discussions with the vendor regarding specifications for specific flavors, a number of revisions were agreed upon. These changes have been incorporated into the final specification.

Support to the Flavor Center continues as regards both flavor specification and the export flavor certification.

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The Blended Dry Flavors program continues with compositions being determined for each of the blended flavors. Specific lots of direct materials are being held for comparison runs at both the Flavor Center and 20th Street. This program will be shifted to the Flavor Center in the future. A current series of comparative analyses are being compiled.

Over 100 individual aroma chemicals were profiled for the chemical components of tobacco and smoke mass spectrometry database being generated.

- C. **Plans:** Continue support to the Flavor Center on an as needed basis, issue the completed Direct Materials specifications, and continue the visits with key vendors on technical issues.

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PROJECT NUMBER : 1902  
PROJECT TITLE : Tobacco Microbiology  
PROJECT LEADER : D. M. Teng  
WRITTEN BY : D. K. Chadick  
PERIOD COVERED : July, 1991

## I. TOBACCO MICROBIOLOGY

A. Objective: To develop methods and to evaluate the microflora in tobacco materials.

B. Results:

### 1. Evaluation of the SOP for Yeast Enumeration

The effects of the addition of varying concentrations of Oxgall to HC agar (HCA) were investigated for its ability to reduce the size and/or number of mold colonies in a mixed population of molds and yeasts. A mixed population of actively growing yeast (R. glutinis) and mold (A. versicolor) was plated on HCA with 0, 1, 1.5, 2, 2.5, 3, 3.5, and 4% Oxgall. No effects were observed from the growth on the plates with Oxgall, regardless of its concentration, when compared to the control plates (1).

### 2. Evaluation of the SOP for Mold Enumeration

Spore suspensions of A. niger were heated at 50°, 65°, and 80°C for 30 minutes and plated on Potato Dextrose Agar (PDA) to determine their heat tolerance. This could be useful in the separation of mold spores and mycelia. After the incubation period no growth was seen on the plates from the spores held at 65° and 80°C. Growth was obtained from the spores held at 50°C; however, the count was slightly lower compared to the unheated, control spore suspension (2).

### 3. Microbiological Support

Burley top coat (BUTC) samples with varying levels of ethanol were submitted by Flavor Technology personnel and analyzed for total microbial counts. No bacterial, mold or yeast growth was observed from BUTC samples prepared at the OC after seven days of storage at room temperature (3).

Studies were initiated to determine the growth characteristics of a yeast culture, Candida utilis, as requested by the Tobacco/Smoke Relationships Project personnel.

A control and three month storage sample of St. John's Bread liquid flavoring was microbially analyzed as requested by Flavor Technology personnel. The samples contained <10 and <100 bacteria/ml at time zero and after three months, respectively. No mold and/or yeast growth was detected from either sample regardless of storage time (4).

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- C. **Plans:** (1) Issue appropriate memos. (2) Continue experiments to establish a method for counting mold mycelial growth vs. mold spores in a mixed population.

D. **References:**

Gaines, O. Notebook No. 9093, pp. 37-38.

Chadick, D. Notebook No. 9044, p. 44.

Chadick, D. Notebook No. 9044, p. 43.

Jones, J. Notebook No. 8590, p. 165.

## II. **CONTROLLED TOBACCO SPOILAGE**

- A. **Objective:** To spoil tobacco under controlled conditions and determine changes in low molecular weight acids and total reducing sumgars produced by mold growth.

- B. **Results:** An additional DBC bright tobacco experiment was conducted. Samples of DBC bright (n=2) were stored at 97% RH and 26°C and allowed to spoil. A portion of the tobacco was analyzed immediately after visible growth was observed (6 days). The remainder of the tobacco was held under the storage conditions an additional six days, 12 days total storage. No increases were seen in low molecular weight acids i.e. citric, malic, oxalic, lactic, propionic, or butyric; however, decreases of 80% and 60% were observed in formic and acetic acids, respectively, in the moldy tobacco (day 6). A 40% decrease in reducing sugars was also seen from the just visibly molded tobacco (day 6) as well as in the tobacco held for a total of 12 days. Some changes were also observed in the CO<sub>2</sub> evolution pattern in the moldy tobaccos compared to the control samples. Odor changes, a fermentation-type odor, were detected only 24 hours after the mold contamination was visibly noticed at day 6 (1).

- C. **Plans:** Changes in low molecular weight acids and total reducing sugars in Oriental tobacco will be investigated.

D. **Reference:**

Weissbecker, L. Notebook No. 9060, p. 24.

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PROJECT NUMBER : 1904  
PROJECT TITLE : Tobacco Physiology and Biochemistry  
PROJECT LEADER : D. J. Ayers/G. J. Patskan  
WRITTEN BY : J. L. Lyle  
PERIOD COVERED : July, 1991

## **I. LOW NICOTINE STUDY**

- A. **Objective:** To investigate the biochemistry of the nicotine biosynthetic pathway and use this information to generate tobacco plants with reduced nicotine levels.
- B. **Results:** Nine batches of roots from Group 29 hydroponically grown Burley 21 tobacco plants have been processed through the 40-65% ammonium sulfate stage. These plants were harvested 3 days after topping. Root extracts were low in enzyme activity and in protein concentration. Specific activities (S.A.) ranged from 10.3-45.5 nmoles/mg protein (units) at 30°C for 30 min. These activities were similar to the activities obtained from roots of plants harvested in past summers. A study is currently in progress in the greenhouse to correlate the outside air temperature to temperatures in the greenhouse and the hydroponic solution (1).

Ammonium sulfate stage samples were pooled and fractionated on two 500 ml bed volume phenyl-Sepharose columns. The specific activity for the peak PMT samples of the first column was determined to be 132.5 units (a 7.5 fold difference in purification compared to the ammonium sulfate fraction). The second column is currently being processed (1).

Ammonium sulfate samples (PMT 242) were analyzed on the RF3 isoelectric focusing unit. Based on the results of these two analyses, no additional ammonium sulfate fractions will be examined on the RF3 unit (2).

Experiments have been conducted for the optimization of the liquid renaturation procedure. Inclusion of dialysis buffer/DTT washing prior to incubation with Extracti-Gel and putrescine appears to enhance renaturation. Renaturation was possible after the starting material was denatured by the addition of Seprazol and boiling after 2 and 5 minutes (3).

Analysis by HPLC of derivatized renatured material subjected to the enzyme assay confirmed the presence of two radioactive peaks, with one corresponding to NMP (3,6). Extracti-Gel does not appear to act as substrate with phenyl-Sepharose material (6). Tris buffer was determined to be the best buffer compared to Tricine, Bicine, EPPs, or sodium bicarbonate for the enzyme assay (4,6).

Work is in progress using HPLC to resolve the components which make up the 60 kD molecular weight region (5).

Two differentially expressed root specific tobacco cDNA clones were digested with EcoRI/XhoI to determine the size of the clone insert. The restriction digests were Southern blotted for further study (7). Several oligonucleotides were synthesized in-



house for anti-sense studies. A relatively quick method to purify oligonucleotides in-house was developed. Several oligonucleotides were purified by this method and then used for cloning and Polymerase Chain Reaction (PCR) work. Restriction mapping of various tobacco root clones was conducted to yield materials for sense and antisense cloning technique development (8). A commercially available plant vector, pBI121 (Clontech), was modified to facilitate anti-sense cloning of tobacco root cDNA insert. The glucuronidase gene (Gus) was cloned into a pBluescript vector. Studies are under way to confirm and characterize these constructs (9,10).

Burley 21 protoplasts were electroporated at varying voltages in the presence of pHIL101 plasmid which confers kanamycin resistance and  $\beta$ -glucuronidase (GUS) activity. In addition DNA prepared from a root-specific clone (PR17) was also electroporated into the cells. These cells are being cultured prior to assay for Gus and/or kanamycin selection. In a preliminary experiment Agrobacterium tumefaciens st. 4404 was electroporated under several conditions in the presence of pBI121 plasmid (which also confers kanamycin resistance and Gus activity). The electroporated bacteria have been plated on kanamycin-containing medium in order to select transformants (11).

- C. **Plans:** Continue to process Group 29 tobacco roots into ammonium sulfate fraction, and continue to prepare PMT samples for the program. Continue studies on PMT renaturation. Continue evaluation of PMT enzyme assay protocol. Continue work with HPLC to resolve 60kD protein. Continue cultures of electroporated Burley 21 cells. Continue to obtain amino acid sequence of fragments of the putative PMT enzyme. Continue to accumulate DNA sequence data for tobacco root overly expressed clones. Continue to sequence PCR clone.

D. **References:**

1. Lyle, J. Notebook No. 9077, p. 34.
2. Davies, S. Notebook No. 9103, p. 58.
3. Turner, D. Notebook No. 9094, p. 112.
4. Steele, M. Notebook No. 9068, p. 44.
5. Yu, T. Notebook No. 9002, p. 139.
6. Nakatani, H. Notebook No. 8384, p. 189.
7. Bower, P. Notebook No. 9032, pp. 76-91.
8. Wahab, S. Notebook No. 8130, pp. 81-82.
9. Malik, V. Notebook No. 8974, p. 122.

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10. Michalik, T. Notebook No. 9109, p. 79.
11. Vaughan, B. Notebook No. 8948, p. 141.

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PROJECT NUMBER : 2500  
PROJECT TITLE : Inorganic Synthesis  
PROJECT LEADER : K. F. Podraza  
PERIOD COVERED : July, 1991  
WRITTEN BY : A. Kallianos

**I. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES**

(Fournier, Kallianos, Paine, Secor)

A. **Objective:** To develop inorganic materials for novel applications for reduced sidestream smoke, for burn-rate modification, enhanced subjectives in cigarettes and for required properties in novel smoking articles.

B. **Results and Plans:** Smoking results were received for a set of cigarettes made with papers containing a blended filler of 5-10% MM  $\text{CaCO}_3$  and 25-20% of a sample consisting of approximately 60% hydromagnesite and 40%  $\text{Mg}(\text{OH})_2$ . This is an aged sample from the reaction of magnesium acetate with potassium hydroxide/carbonate. No difficulties were observed in the handsheet making of this sample. A sidestream smoke reduction of 66% was exhibited with a static burn time of 9.9 minutes and good ash characteristics. Subjectives are pending.

Five handsheets, each containing a different magnesite sample; 1) 98.5% magnesite/1.5%  $\text{Mg}(\text{OH})_2$ , prepared by the "high pressure" hydrothermal reaction of  $\text{Mg}(\text{OH})_2$  with  $\text{CO}_2$ ; 2) 78% magnesite/22%  $\text{Mg}(\text{OH})_2$ , also prepared by the "high pressure" hydrothermal reaction of  $\text{Mg}(\text{OH})_2$  with  $\text{CO}_2$ ; 3) 85% magnesite/15%  $\text{Mg}(\text{OH})_2$ , prepared by the "low pressure" hydrothermal treatment of hydromagnesite; 4) 99+% magnesite, prepared by the "low pressure" hydrothermal reaction of hydromagnesite with potassium bicarbonate; 5) a milled (MCS) Baymag magnesite, were prepared and submitted for cigarette making and subjective comparison. These samples are being evaluated for synthetic magnesite scale-up.

A technical package on the different synthetic routes of magnesite, including RSA's results and the coupon experiments, has been compiled for Pressure Chemical Company in Pittsburgh. Seven commercial hydromagnesite samples were submitted to Analytical for trace metals analyses. The purpose of this is to source a commercial hydromagnesite for the scale-up work.

A sample consisting of approximately 34% hydromagnesite, 27%  $\text{Mg}(\text{OH})_2$ , and 39%  $\text{CaCO}_3$ , prepared by the reaction of a mixture of magnesium acetate and MM  $\text{CaCO}_3$  in water with a potassium carbonate/hydroxide solution, was submitted for handsheet making. A request for three magnesite/calcite bilayer handsheets was made; the bottom layers consisting of a 40% fill of magnesite at a 20, 30, and 40  $\text{g/m}^2$  basis weight and the top layer in each case a 30% fill of MM  $\text{CaCO}_3$  at 15  $\text{g/m}^2$ . The purpose of the magnesite/calcite bilayer handsheets is to alleviate the streaking problems associated with present magnesite models without adversely effecting the sidestream reduction properties. A sample of hydromagnesite prepared in an aqueous propylene glycol solvent system has also been submitted for handsheet making. This sample consists of spherical particles with a narrow particle size distribution.

Handsheets prepared from the replicate preparation of a promising candidate from the non-sol-gel procedure, which is being considered for scale-up synthesis, have been analyzed and submitted for cigarette making and smoking evaluation. Preparatory to our contracting for the scale-up synthesis of this material, W.B. Edwards requested some additional information on the reaction as it is currently being carried out. He further suggested the use of bulk starting materials. These requests have been communicated to Dr. Schleich and samples of the bulk starting materials have been shipped to him at N.Y. Polytechnic for evaluation.

Several samples of mag carbonate aggregates prepared via non-sol-gel as well as aqueous sol-gel routes have been submitted for SEM and electron diffraction examination in further attempts to determine unique compositional features, which may support composition of matter claims in patent applications currently in preparation.

The inverse relationship between sedimentation rate of the particles and surface area, observed and preliminarily reported previously on three samples, has been realized for all nine samples from the 3x3 matrix of experiments varying reaction temperature and pH. Three samples from this series have been selected for replicate syntheses to reconfirm the above relationships and to obtain sufficient quantities of materials for handsheet making, in order to evaluate the role of sedimentation rate relative to paper-making facility. The samples will serve as models in mechanistic studies for evaluation of filler properties in sidestream smoke control.

Eight samples of commercially ground limestone products were received. They have been repackaged and submitted for handsheet making, Analytical Microscopy, and miscellaneous testing. Three of these are ground dolomites and should definitely establish whether natural dolomite has any interest for us. Dolomite is difficult to synthesize, especially in the highly ordered form typical of the natural material. Five of the samples are calcites, derived from the grinding of natural limestones or marbles. These samples, in addition to the three ECC and J.M. Huber samples submitted previously, should give us a good indication of the suitability of natural calcites for use as "rhombohedral" morphology calcite.

Cigarette models prepared with fillers from the coprecipitated magnesium/calcium carbonate/hydroxide program achieved sidestream reductions in the range of 25 to 62%, with static burn times ranging from 6.5 to 8.1 minutes; those with reductions better than 40% (six of the models) were evaluated subjectively and judged to be moderately acceptable. Subjectives were also received for the models containing northupite  $[\text{Na}_3\text{Mg}(\text{CO}_3)_2\text{Cl}]$  or tychite  $[\text{Na}_6\text{Mg}_2(\text{CO}_3)_4\text{SO}_4]$ ; these were sufficiently low as to discourage further interest in either.

X-Ray diffraction powder patterns were received for three samples. The two samples from the high-temperature carbonation of portlandite-brucite mixtures in the absence of calcium chloride proved to be the desired blend of calcite and hydromagnesite. Handsheets prepared from these samples have been submitted for elemental analyses and cigarette making and smoking. The product from the synthesis of rhombohedral calcite via low-temperature reaction of calcium chloride with potassium bicarbonate proved to contain minor vaterite in addition to the calcite.

#### Structures of Substrates

calcium carbonate, calcite, aragonite  $[\text{CaCO}_3]$

hydromagnesite  $[\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}]$

magnesite  $[\text{MgCO}_3]$

magnesium hydroxide, brucite  $[\text{Mg}(\text{OH})_2]$

portlandite  $[\text{Ca}(\text{OH})_2]$

## II. MISCELLANEOUS: (Howe, Secor, Paine)

**Pseudooxynicotine Dihydrochloride.** A sample of trans-nicotine N'-oxide, 1.61 g, was reacted with acetic anhydride until no starting material remained by TLC. The dark solution was made alkaline, extracted with chloroform, dried (sodium sulfate), evaporated, and chromatographed on 10 g of silica gel to provide 1.10 g of light yellow oil, pseudooxynicotine N'-acetate (N'-Ac) was confirmed by IR. One gram of N'-Ac was treated with 5M sodium hydroxide (30 ml) and steam distilled to a receiver containing dilute HCl. Evaporation of two fractions collected gave 104 mg and 340 mg, respectively. NMR and TLC indicated a mixture of pseudooxynicotine and some other component. A sample recrystallized from alcohol-ether gave one spot on TLC and a PMR spectrum that matches the standard and the literature spectrum.

A sample of six milligrams (6 mg) of N'-Ac was steam distilled from aqueous base as above to a receiver containing dilute HCl. The solvent was evaporated to give pseudooxynicotine, confirmed by NMR, and one impurity as above, 11.5 mg.

(2S,4S)-1-methyl-2-(3-pyridyl)-4-[[3-carboxypropanoyl]-oxy]-ethylpyrrolidine. A previously prepared impure sample of the material was partially purified on a chromatotron and further purified by R. Izac using HPLC. In addition, a further 700-750 mg sample of this material was prepared starting with impure (3-t-butoxycarbonylmethyl)-1-methyl-5-(3-pyridyl)-2-pyrrolidinone. Final purification of this material will be carried out when needed by Project 6902 for the antigen binding project.

**Calcium Menthyl Carbonate.** Calcium metal was converted to calcium methoxide, under anhydrous conditions, and suspended in anhydrous toluene. Menthol was added, and the methanol distilled off. There resulted a solution/suspension of calcium menthoxide in toluene. This was treated with a stream of carbon dioxide. The pH of the medium fell with time (after quenching aliquots with water), suggestive of a reaction

taking place. However, carbon-13 NMR investigation of the supernatant solution showed peaks for only toluene and menthol. There was no detectable quantity of carbonate or menthylcarbonate in solution. It is doubtful that the target molecule is stable enough for our needs.

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PROJECT NUMBER : 2501  
PROJECT TITLE : Smoke Chemistry  
PROJECT LEADER : D. D. McRae  
PERIOD COVERED : July, 1991

## **I. SIDESTREAM SMOKE CHAMBER**

- A. **Objective:** Operate an environmentally controlled chamber to measure selected components of sidestream smoke.
- B. **Results:** Chamber runs in support of BCR efforts have been completed. Memos reporting the results for the cigarettes from the sidestream analytical-subjective series (X8D1WG - X8D1WK) smoked under static conditions have been issued.

In preparation for the subjective portion of the sidestream analytical-subjective study a number of modification and maintenance items have been addressed. The air conditioning system was serviced to reduce the temperature and humidity swings associated with the cycling of the equipment. The smoking machine was serviced to try to eliminate occasional loading problems. New face masks have been made for the sniffing port. New sampling lines were run from the chamber for the mass spectrometer and other sampling lines were modified to allow the control and measurement of the flow rates.

All analyses for the nicotine recovery study have been completed and the data is being analyzed. Initial results indicate that nicotine was not present in the chamber before the first run of a day. The chamber is clean at this point having been washed down at the end of the previous day. Nicotine was also not present between runs and after the last run of the day. In both of these latter cases the smoke in the chamber was evacuated but the chamber was not washed. Nicotine carryover from run to run does not appear to be a problem.

Examination of the particle size distributions from a number of cigarette samples has shown that the mean particle diameter decreases as the amount of sidestream smoke generated also decreases. This observation led to an analysis of the correlation between the various mass- and visibility-based methods for measuring sidestream smoke. In general, all of the methods agree remarkably well considering the widely different techniques. Some techniques may over- or underpredict the sidestream reduction values but more data needs to be collected for an accurate analysis.

- C. **Plans:** The subjective portion of the analytical-subjective study is scheduled to begin immediately with tests to determine the optimum smoke concentration and our ability to measure analyticals at this level. Development of the analytical procedure for acrolein is continuing.

## II. SIDESTREAM SMOKE

- A. **Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream semivolatiles and gas phase; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.
- B. **Results:** The Japanese cigarette study using the prototype CORESTA smoking machine has been completed and the data will be analyzed shortly. Mainstream and sidestream TPM and puff count data were generated in an effort to determine why Japanese cigarettes appear to produce less sidestream smoke.

The desorber/gc/ms is now being used to measure sidestream smoke from the chamber. A careful study of the levels of the compounds found in the laboratory and chamber air has been started to provide information on the amounts and types of these background materials for comparison with sidestream smoke. Over one hundred volatile compounds were identified in normal laboratory air during an 18 hour collection. Most of these compounds are present in ppb levels and have been reported previously in the scientific literature. While some of the compounds found are present in cigarette smoke, most come from other common sources such as gasoline and diesel fuel and exhaust, cleaning compounds and emissions from paint, carpet, etc. Analyses of sidestream smoke and air (no smoke) from the chamber exhibit some problems. Levels of sidestream volatiles for no smoke, 2, 4 and 6 cigarettes were much larger than for laboratory air but were not sized according to the number of cigarettes smoked. Sampling techniques continue to be studied and a number of modifications have been made to improve them including better flow control, different collection times and the possible use of an internal standard. A musty odor present in the chamber has been tentatively identified as 2-ethyl-1-hexanol, a starting product for the ubiquitous plasticizer, dioctyl phthalate. Minor changes in the chamber purge procedure are being considered.

- C. **Plans:** CORESTA smoking of the cigarettes for the Aromatek 150 distribution study will be performed as time permits during the sidestream subjective study. Optimization of the gc/ms system is continuing.

## III. MISCELLANEOUS

- A. Neutron radiography - The rod uniformity of cigarettes produced by an experimental maker are being measured along with IM13 cigarettes as a control. Modifications of the data collection program for measuring mass burn rates and the deposition of condensibles are complete.
- B. Pyrolysis gc/ms analyses at 300°C have been performed on a variety of samples from different requestors. Product identifications are in progress. In one case, the

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pyrolyzed compound did not release the desired product, thus, no further work is planned on this sample. DCM extracts of glycerin and triacetin have been submitted for analysis by electron impact and chemical ionization mass spectrometry. Memos have been issued to the requestors concerning previous work.

- C. The low molecular weight gases of mainstream smoke are being studied. Three sulfur containing compounds thought to be  $\text{SO}_2$ , COS and  $\text{H}_2\text{S}$  are present but are not separated from the much larger CO and  $\text{CO}_2$  peaks.

PROJECT NUMBER : 2520  
PROJECT TITLE : Organic Synthesis  
PROJECT LEADER : Y. Houminer  
PERIOD COVERED : July, 1991

## I. FLAVOR RELEASE TECHNOLOGY

- A. **Objective:** To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.
- B. **Results:** A series of experiments were conducted with PolyMIC to determine effective levels for plasticizers. PolyMIC was slurried in abs. ethanol to which was added the target level of the test plasticizer. Material was produced with triacetin and triethyl citrate at 5 and 10%. After removal of the solvent, the product was a free flowing powder in all cases. Each of the treated PolyMIC samples showed a lower melting point, approximately 10 and 20°C for 5 and 10% respectively. The results of these experiments were given to W. Nichols who conducted a 2 pound experiment with triacetin at 10%. The resulting material was successfully applied as a hot melt to cigarette paper during cigarette manufacture.

The reaction of  $\alpha$ -hexylcinnamaldehyde and cinnamyl isobutyrate gave the desired  $\beta$ -hydroxyester. A small sample was purified and was shown to undergo smooth pyrolysis at 300°C to give the corresponding aldehyde and ester.

Studies on the acetalation of  $\alpha,\alpha$ -trehalose with  $\alpha$ -hexylcinnamaldehyde continued this month. Preparative HPLC isolation of products from the reaction of trehalose and  $\alpha$ -hexylcinnamaldehyde dimethyl acetal yielded fractions which contained significant quantities of  $\alpha$ -hexylcinnamaldehyde and some trehalose. It was concluded that breakdown of the desired products was taking place on the silica HPLC column.

CR-2950 was incorporated into two different cigarette papers at a level of 1.5 - 2.0% by Ms. S. Tafur. Handmade cigarettes were placed in desert and jungle rooms for 4 days. Analysis of the cigarette papers by a reversed phase HPLC procedure showed no difference between the amount of CR-2950 on paper of the test cigarettes and the control.

Hydrolysis of menthyl 3,4:5,6-di-O-isopropylidene-D-gluconate was carried out under two different conditions. We have isolated menthyl 3,4-O-isopropylidene-D-gluconate, which on pyrolysis via a 220° GC injector port gave significant amount of menthol. We are continuing our effort to isolate menthyl D-gluconate.

In an attempt to prepare bis-(3-oxo- $\alpha$ -ionyl)carbonate through the intermediate 3-oxo- $\alpha$ -ionyl chloroformate, three compounds were isolated and identified, none being the desired product. Two of the compounds were identified as Z- and E-megastigma-4,6,8-trien-3-one, and the other as 3-oxo- $\alpha$ -ionyl chloride. All three compounds are decomposition products of 3-oxo- $\alpha$ -ionyl chloroformate.

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## II. FILTER MATERIALS AND PAPER TECHNOLOGY SUPPORT

- A. **Objective:** To find outside suppliers for large quantities of new filter materials and inorganic paper additives.
- B. **Results:** Jerry Johnson and C. Woodings of Courtaulds visited the center and extensive discussions were held covering current work and samples status. New handsheets have been made from Courtaulds partially deacetylated cellulose and yielded filters with acceptable RTD. These will be attached to cigarettes and subjective evaluation conducted shortly. Discussions with Courtaulds have indicated that the problems with dispersion of the cut CA is probably due to the finishing agents. Plans have been formulated to evaluate alterant agents to allow for effective dispersion.

Trials conducted at Nina mill have shown that Courtaulds' fibrids can be effectively incorporated into CA and CA/soft wood mixtures to give acceptable paper. Problems were encountered with craping of the paper. Discussions were conducted with Courtaulds on this and an additional 50 kg of fibrids has been requested for trials.

## III. MISCELLANEOUS

Synthesis of delta-Cadinene: 4-Methylanisole was reduced by lithium/ammonia to give 4-methyl-3-cyclohexenone. This was converted to the pyrrolidine enamine for reaction with 6-methyl-4-hepten-3-one obtained by condensation of 2-butanone with isobutyraldehyde. The enamine and 6-methyl-4-hepten-3-one were heated under reflux in toluene for 4 days. Preliminary analysis of the complex crude reaction product indicated some delta-cadinenone was formed. Following isolation, this will be reduced in two steps to delta-cadinene.

The total synthesis of delta-cadinene is reported to give an isolated yield of less than 1%. This is less than the amount of delta-cadinene in Cade Oil. Accordingly, isolation by preparative gas chromatography is being carried out.

2022143199

**PROJECT NUMBER :** 2526  
**PROJECT TITLE :** Greenhouse Operations  
**PROJECT LEADER :** R. T. Bass  
**PERIOD COVERED :** June-July, 1991

**I. GREENHOUSE STUDIES AND RESEARCH ACTIVITIES**

(R. Bass, G. Newell, G. West)

- A. Objective:** Maintain the R&D Greenhouses, conduct plant research studies, provide greenhouse-grown tobacco materials for support of other R&D programs, and provide requested assistance for special projects.
- B. Results:** The regular Greenhouse plant production activities have been completed, including the preparation of 770 gallons of nutrient solution, five insecticide applications, the performance of all cultural practices, and the seeding and transplanting of Burley 21, K-326, Speight G-28, Wernsman's NC Air-cured Aromatic NC-37-1, N. rustica ACC-48, N. rustica R-120, and Dryopteris filix-mas ferns.

The regular Greenhouse operations and maintenance jobs have been done on schedule including the cleaning of the KOOL-CEL water storage tank and the evaporation pads, lubrication of the side vents, and installation of the shade cloth for the summer season.

The production and harvest of the 52 hydroponically grown Burley 21 plants for Group 29 has been completed. The total wet weight yield of fresh root material was 22,306 grams with an average per plant yield of 429 grams. The 52 plants for Group 30 have been transplanted into the hydroponic system. These plants are showing good color and growth at this time.

As requested by M. Zimmermann, a fresh supply of Dryopteris filix-mas fern rhizomes were obtained from Blue Ridge Nursery. Thirty-five of these have been potted and placed on the benches where they are now sprouting and showing young leaves.

The group of eighteen mature Solanum dulcamara plants were harvested and provided for M. Zimmermann in order for the leaves to air dry for processing.

The fruit (strobiles) from several remaining hop plants were harvested and provided to Dr. P. Bower for research use.

The seed for two strains of Nicotiana rustica var. brasilia, namely ACC. 48 and R-120, have been obtained from Dr. V. Sisson of the U.S.D.A. at Oxford Tobacco Research Station. These have been seeded and transplanted in an effort to obtain a seed quantity increase. These seeds have been requested for use by Project 1904.

Greenhouse tours have been provided as requested for various visitors including PM International and Leo Burnette Advertising Agency.

At the request of and under the supervision of the R.S.O., A. Frisch, our stored inventory of chamber grown  $^{14}\text{C}$ -tobacco leaf materials was transferred from plastic containers into two new air-tight Nikko cabinets. These cabinets will provide improved lockable storage conditions for the  $^{14}\text{C}$ -labelled leaf material samples.

During May and early June we have maintained a vigorous effort to prepare the entire  $^{14}\text{C}$ -Plant Growth Chamber System for Run Number 14 (actually 13 but we hesitate to use that number). Earlier this year we planned to conduct this chamber run to grow tobacco plants for the production of  $^{14}\text{C}$ -labelled solanesol. All of the component parts of the system were made ready and the system tested for operation in early June. On June 10 the four transplant size plants of NC-37-1 were placed in the chamber, the door sealed in place and the system put into operation. After several days to allow the plants to acclimate, the injection of  $^{14}\text{CO}_2$  was begun on a demand basis. The system has been in continuous operation for 40 days with the  $\text{CO}_2$  injections increasing from 3 to 22 per day. The plants have grown to a height of 26 inches showing 24 leaves per plant. The upper leaves do show some effect of the  $^{14}\text{CO}_2$  radiation atmosphere. At present we have recorded a total  $^{14}\text{CO}_2$  injection time of 2302.0 minutes for 544 injections. As "cold controls" we are also growing some NC-37-1 plants on the Greenhouse bench as well as outdoors.

C. **Plans:** Greenhouse operations and maintenance will be continued along with all requested support activities.

D. **References:**

1. G. Newell, N.B. No. 9090, 7662, 9113
2. G. West, N.B. No. 8559
3. R. Bass, N.B. No. 8999, 9113

## II. **COOPERATIVE TOBACCO VARIETY EVALUATION PROGRAM**

(R. Bass, G. Newell and G. West)

A. **Objective:** To participate in the cooperative tobacco industry program in order to assure that the quality of all new tobacco varieties meets or exceeds all requirements as defined by minimum standards program.

B. **Results:** The combined Clemson Tobacco Tour and the South Carolina Tobacco Variety Test Tour were attended on July 9 and 10. Included in the tour were visits to the Pee Dee Tobacco Research Station, to cooperative grower test field plots for disease control, fertilizer management, sucker control, nematode control, and the Regional Farm Variety Test. The North Carolina Tobacco Tour was attended on July 11 and included visits to the Whiteville Tobacco Research Station and several Regional Farm Variety Tests located in the Border and Eastern tobacco belts. Inspection of the variety test plots showed all entries in the Regional Farm Test and the Regional Small Plot Test to be genetically stable. The appearance of the plots

and the general crop in these areas is good with a potential for good quality. The rainfall has been somewhat variable, but adequate in most areas.

As requested by Dr. W. Hempfling, arrangements have been made with personnel of the Research Station at Blackstone, VA. to provide six field grown mature plants of variety Burley 21. These plants will be harvested and sampled at the appropriate time.

Information concerning the resistance level of certain Burley tobacco varieties to TMV has been provided as requested for Drs. P. Bower and B. Davies.

The plans and preparations for the 1991 PM R&D Tobacco Tour are in progress. The tour is usually held during August, depending on the growing season and the Henderson market opening date.

A trip was made with H. Grubbs to N.C. State University for research discussions concerning data and analyses with regard to total secondary alkaloids and solanesol.

As requested, we provided tobacco material samples to the Analytical Research Division and to Project 6908 for their use. One sample was Oriental leaf, grade TIU, 1987 crop, mark 211, and the other was 10 fresh green leaves of mid-stalk position from Greenhouse hydroponically grown Burley 21 tobacco (Group #29).

C. Plans: To continue to participate in the Cooperative Tobacco Variety Evaluation Program.

D. References:

1. G. Newell, N.B. No. 9090
2. G. West, N.B. No. 8559
3. R. Bass, N.B. No. 8999

**PROJECT NUMBER :** 2705  
**PROJECT TITLE :** Tomorrow  
**PROJECT LEADER :** R. W. Dwyer  
**PERIOD COVERED :** July, 1991

**I. DESIGNING LOW-MBR CIGARETTES**

- A. **Objective:** Evaluate the effects of cigarette design parameters on mass burn rates.
- B. **Status:** We have evaluated the results of a wrapper study in which the chalk level, basis weight, and permeability were varied. These wrappers were used to make cigarettes in which the blend, tobacco weight, and filter design were held constant. We evaluated the effects of the wrapper variables on mass burn rates and puff counts. The results show that the MBR and puff counts are most affected by the chalk contents and permeabilities of the wrappers and are unaffected by the amount of fiber present. The results also suggest that the chalk level plays a greater role in affecting MBR and puff count than does permeability. All of the results evaluated to date were from samples containing Albacar chalk.
- C. **Plans:** We are waiting for results on cigarettes made with wrappers containing Multifex chinks at various levels, and different types and levels of burn additives.

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PROJECT NUMBER : 2706  
PROJECT TITLE : New Expanded Tobacco  
PROJECT LEADER : T. M. Howell  
PERIOD COVERED : July, 1991

## **I. TOBACCO ORDERING AND COLLAPSE**

- A. **Objective:** Evaluate the reordering of NET product by exposure to conditioned humid air.
- B. **Results:** Testing of the Frigoscandia Spiral Conveyor was continued and the runs to date have demonstrated the ability of the unit to reorder NET tower exit product from about 3% OV to 11.5% OV. Results from the laboratory environmental chambers show that a wide range of temperatures and air velocities may be used to reorder the NET product in the unit without suffering significant losses in tobacco CV.
- C. **Plans:** Testing will continue to establish the optimum operating conditions for humid-air reordering in the Frigoscandia Spiral Conveyor.

## **II. THERMAL STABILITY OF NET IMPREGNANT**

- A. **Objective:** Determine the temperature range of impregnant release from gaseous CO<sub>2</sub> impregnated tobacco as a function of filler OV using the hot oil immersion technique.
- B. **Results:** Feedstocks with target OVs of 12%, 15%, 18% and 21% were impregnated with gaseous CO<sub>2</sub> in D Pilot Plant. Aliquots of the impregnated filler were taken for tower expansion, oil immersion and oven expansion experiments. The tower expanded portions were submitted for SV, CV and %OV (equilibrated and unequilibrated) analysis. Preliminary results indicate that the 15% OV filler had the highest SV and CV. Only the impregnant in the 12% OV material appeared to be stable at temperatures above 0°C. Except for the 12% OV material, the impregnated fillers were no longer expandable after being subjected to temperatures below 0°C. using the hot oil immersion test.
- C. **Plans:** Experiments have been designed to test the effects of time on the thermal stability of the impregnant using the hot oil immersion technique. A report of the procedure and the data collected is being prepared.
- D. **References:** Notebook no. 8309, pp 182-190.

## **III. SUPERCRITICAL FLUIDS EXTRACTION**

- A. **Objective:** Assist Kraft General Foods in exploring the application of supercritical carbon dioxide to the extraction of fat from cocoa and other fat containing food products.



- B. **Results:** The extraction conditions for removing the fat from cocoa were selected and several runs were made in order to produce product for evaluation by KGF in Dover, Delaware. Similar conditions were used to extract the fat from various coconut products. To date, weight changes after extraction are showing fat reductions in the 80% range for raw and toasted coconut. Although the residual fat content of the coconut is about 20% to 30%, standard pressing techniques generally do no better than 40% residual fat.
- C. **Conclusions:** Supercritical carbon dioxide appears to be a viable method for removing fat from coconut as well as from cocoa. Slower let down procedures are required for the coconut in order to prevent breakage as the CO<sub>2</sub> escapes.
- D. **Plans:** Various peanut products were received from Glenview and will be evaluated for fat extractability using supercritical CO<sub>2</sub>.

2022143205

**PROJECT NUMBER :** 2707  
**PROJECT TITLE :** Vision Inspection Technologies  
**PROJECT LEADER :** R. J. Maher  
**PERIOD COVERED :** July, 1991

## **I. PACK INSPECTION SYSTEM**

- A. **Objective:** Develop and implement an on-line cigarette pack inspection system with the capability for global inspection.
- B. **Results:** The QA force feed test was done this month. This test was designed to obtain the inspection efficiencies of the OSIRIS system, the CI-750 (3 camera Itran) and the MVP (2 camera Itran). The OSIRIS system had been trained using packs from packers 93 and 94. No operational difficulties were encountered during the test. It was necessary to perform one adapt in order to reduce a high number of nuisance rejects observed during the first pass. QA is planning to report the test results in August.

The computer hangs with the OSIRIS system is a result of the software. A new release of the microcode has been received from Matrox and will be tested on the laboratory system.

Progress has been made on the incorporation of the warp code into the current operating software. This effort will simplify the definition and modification of both the pack gauge overlay and the inspection zones.

The support effort required by Itran in their development of the FS-Osiris system has remained minimal.

- C. **Plans:** Test the most recent Matrox software release in order to eliminate the computer hangs. Complete the incorporation of the warp code into the current system. Begin to incorporate the code that will periodically sample a good pack and automatically incorporate the information into the inspection filter.

## **II. PRINT WEB INSPECTION**

- A. **Objective:** Develop a system for the global inspection of print web on the printing press.
- B. **Results:** Preparations for building the prototype on-line web inspection system are in progress. Because of CHP shutting down for 3 months for HVAC renovations, the definition of the prototype system has changed. It is now felt that the best system to place at CHP will be a 4 camera system. This system will be used to both characterize the CHP web and to demonstrate the inspection capability.

The DataCube Maxvideo20 board and the Maxscan 10MHz board have been installed in the Sun4 computer in the laboratory. This imaging system has been used with an RS-170 array camera in order to verify correct operation. Some problems have been uncovered and a solution is being sought via DataCube.

The second Dalsa TDI camera has been received and has been successfully tested.

Work continues on the design of the explosion-proof enclosures with the help of Development Engineering.

Discussions continue with vendors that claim 100% web inspection capability. Eltromat will present their system at the PRINT '91 Conference in September. Representatives from Presco Technology are planning to discuss their system at PM during the month of August.

- C. **Plans:** Begin the integration of the hardware for the prototype system. Identify and acquire suitable explosion-proof enclosures with the help of Development Engineering. Continue to evaluate commercially available web inspection systems.

### **III. OFF-LINE INSPECTION**

- A. **Objective:** Develop and implement a system for the inspection of incoming materials.
- B. **Results:** All components for the two color QA blank inspections system have been ordered. The longest delivery item will be the material handling-motion control system ordered from PST, Inc. Delivery of the material handling-motion control system is slated for October.

The design of the user interface was completed and is currently being reviewed.

Larry Brice from the QA Department has compiled a list of the vendors of the incoming materials that will be inspected by the two color system and has obtained a list of the individual codes. He has prioritized the list of incoming materials that will be inspected. The list was prioritized for inclusion into the inspection system database.

- C. **Plans:** Complete the algorithm implementation and begin coding the user interface.

2022143207

**PROJECT NUMBER :** 6502  
**PROJECT TITLE :** Process Monitoring and Real Time Measurements  
**PROJECT LEADER :** R. W. Kanipe  
**PERIOD COVERED :** July, 1991

### **PROCESS METHODS DEVELOPMENT**

A. **Objective:** To develop a QA procedure for acceptance of adhesive shipments at Cabarrus.

B. **Results:**

Various levels of methanol, formaldehyde, acetic acid, and ammonia were added to selected adhesive samples to evaluate the effects on gas-phase and/or liquid-phase FTIR spectra as compared to reference spectra. These compounds have been identified as typical contaminants in adhesives. Results showed that 1% methanol and 2% acetic acid could be visually detected in the liquid-phase (ATR FTIR) spectra. For the gas-phase spectra, methanol was visually detected at 0.02% for an adhesive without vinyl acetate and 0.1% for an adhesive with vinyl acetate. Acetic acid was visually detected at 8% and 1% for adhesives with and without vinyl acetate, respectively. Ammonia was visually detected at ~0.2% in the vinyl acetate adhesive sample. Results for samples spiked with formaldehyde were inconclusive.

Experiments were completed to determine the stability of the twelve reference adhesives during the development stages of the headspace FTIR method. The samples had aged two months since the initial stages of development. Headspace for three of the adhesive samples did show reduced FTIR absorbance spectra when compared to reference spectra. For two of the samples, the reduction was primarily attributed to excessive sample handling during the two month period. Spectra for the remaining nine adhesives showed no significant differences when compared to reference spectra.

C. **Conclusions:** In general, typical contaminants in adhesives generate spectral differences which are visually detectable at certain levels when compared to reference spectra. Sample integrity was acceptable during the development stages of the headspace method; however, indications are that the adhesives do not have an infinite shelf life and sample integrity may be affected by excessive sample handling.

D. **Plans:** Determine the acceptable level of spectral variation between sample and calibration data. Continue to develop software to facilitate routine sample analysis.

E. **References:**

1. Parrish, M., PM Notebook 9106, pp. 7-12.
2. Lewis, W., Lewis, PM Notebook 9095, pp. 23-28.

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PROJECT NUMBER : 6503  
PROJECT TITLE : Chemical Analysis  
PROJECT LEADER : B. M. Handy  
PERIOD COVERED : July, 1991

### **I. PROJECT TOMORROW**

- A. **Objective:** Support R&D efforts to measure and modify ignition propensity of cigarettes.
- B. **Results:** Fabric samples, containing the burn promoter potassium acetate, supplied by RJR for the CORESTA Collaborative Study were analyzed for potassium by ICP. Analysis of a second group of fabric samples is currently in progress. Banded papers from June 1991 Beloit trials were analyzed for propyl paraben to determine the level of cellulon addition. Laboratory prepared sheets of applied cellulon similarly were analyzed to determine applicability of method.
- C. **Plans:** Complete ICP analysis of fabric samples. Continue support as needed.

### **II. CAST LEAF RCB PROCESS**

- A. **Objective:** Support R&D studies to determine chemical basis of RCB Darkening.
- B. **Results:** Samples of RCB, unheated control and heated and self-heated test samples, were analyzed for acetic, formic, citric, lactic, malic and oxalic acids, chlorogenic acid, rutin, scopoletin, fructose, glucose, sucrose, soluble ammonia, nitrate nitrogen, calcium, phosphorus, pH and OV. Investigation of ion chromatography to determine gluconic acid in RCB is currently in progress.
- C. **Plans:** Continue analytical support for RCB Darkening Study.

### **III. PRIMARY IMPROVEMENT**

- A. **Objective:** Support engineering efforts, Primary Improvement Program, in improving the uniformity of burley spray application by use of a Sagemuller Steam Tunnel.
- B. **Results:** Sieve fractions of strip and lamina passed through a Sagemuller Steam Tunnel installed prior to Burley Spray Cylinder were analyzed for alkaloids, reducing sugars, fructose, glucose, and sucrose to determine the levels of burley spray on the tobacco as a function of particle size.
- C. **Plans:** Continue to support engineering efforts to improve uniformity of burley spray application.

PROJECT NUMBER : 6505  
PROJECT TITLE : Special Investigations  
PROJECT LEADER : D. F. Ingraham  
WRITTEN BY : R. W. Kanipe  
PERIOD COVERED : July, 1991

## I. MENTHOL

- A. Objective: To develop methods for the quantitation of menthol release compounds such as beta-1-menthyl itaconate, monomer and polymer, and POLYMIC. To utilize these methods for sample analysis as required.

B. Results:

A capillary GC procedure was developed for POLYMIC using the response of menthol for quantitation. Menthol was released from POLYMIC by refluxing with an ethanolic/KOH solution. POLYMIC was determined in two filler samples (J1AFU and J1AFV) having a target application rate of 2.7%. The levels of POLYMIC measured in the samples using this method were 2.4% and 2.7% by weight, respectively.

Menthol released from a POLYMIC hot-melt sample was determined by comparison to a POLYMIC "powder" standard. Menthol was released from both samples as described above. The menthol response of the POLMIC hot-melt was 66% of the menthol response for the POLYMIC standard. These results included any free menthol which may have been present in the hot-melt prior to refluxing.

Poly( $\beta$ -1-menthyl itaconate) (POLYMIT) was determined in loose filler (J1AKJ) and corresponding cigarette filler (X6D1BWL) having a target application rate of 4%. The POLYMIT levels measured in these samples were 4.1% and 4.3% by weight, respectively.

- C. Plans: Plans are to analyze samples for such menthol release compounds as required. Methods will be developed as needed including the evaluation of purge and trap techniques.

## II. R&D AND OPERATIONS SUPPORT

- A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

An HPLC method was developed for the determination of glycyrrhizic acid in tobacco in support of Marlboro Standardization. The method combined the sample preparation procedure from R&D Method E-78 (by G. Carter) and the

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chromatographic conditions of Flavor Specifications Method #6 (by M. Zimmermann). Approximately 100 tobacco samples were subsequently analyzed using the modified procedure.

A "mini" training course was provided for five representatives from four tobacco suppliers on HPLC basics and the HPLC determination of methoprene. The suppliers are in the process of setting up the methoprene method in their laboratories for routine testing of tobacco purchased for Philip Morris.

A study to evaluate chemical changes leading to "stale" cigarettes continues. Conditions were established to reproducibly reduce the water content of Marlboro cigarettes from 11.5% to ~9% using vacuum drying, desiccant ( $\text{CaSO}_4$ ), nitrogen sweep, and an oven. Re-conditioning of the cigarettes to bring their water content back to 11.5% is in progress in preparation for subjective evaluation scheduled for July 29.

Jaguar CB10 was examined using Curie Point Pyrolysis followed by GC/MS of the volatile pyrolyzate. The material was pyrolyzed at 315°C and 590°C. The major compound observed at both temperatures was 1-hydroxy-2-propanone. Other compounds observed included ketones and hydroxy compounds. Jaguar CB10 is under evaluation as a potential binder for the new cast leaf process.

Nicotine was determined in XAD tubes through which  $\text{CO}_2$  had been vented from the NET process and also in methanol washes of the corresponding  $\text{CO}_2$  cylinders. Nicotine was also determined in isopropanol washes of vented  $\text{CO}_2$  cylinders from the current DIET process. Data were reported to E. Thomas.

In support of an ethanol accountability study in progress at Cabarrus, a series of traps were evaluated for efficiency and stability of ethanol containment. Ethanol vapors were drawn through two methanol, one silica gel, and two charcoal traps (in series) using a vacuum induced air flow (2 L/min) over the ethanol. Results showed that 97% of the ethanol was trapped in the two methanol traps. A recommendation for trapping ethanol vapors was made accordingly.

Three DBC Bright samples were analyzed for propionic and butyric acids by capillary gas chromatography. None of the samples contained either of the acids which are typically used to monitor spoilage. The limit of detection was approximately 10  $\mu\text{g/g}$  for both acids under the operating conditions specified. This preliminary evaluation was in support of the mold issue.

Fifteen RCB samples were analyzed for butyric acid by capillary gas chromatography. Butyric acid was not detected in any of the samples except one which contained < 50  $\mu\text{g/g}$ . The limit of detection was approximately 10  $\mu\text{g/g}$ . Spoilage was being monitored in these samples as part of the ongoing RCB Stability Program.

Eighteen CEL and SEL samples were analyzed for propionic acid to monitor potential spoilage in the new Weigand Evaporator. Determination was made by capillary gas chromatography. Levels of propionic acid in the SEL samples ranged

from none detected to about 25  $\mu\text{g/g}$ . In the CEL samples, levels ranged from about 60 to 100  $\mu\text{g/g}$ . The limit of detection of propionic acid in the SEL was approximately 10  $\mu\text{g/g}$  under the operating conditions specified.

2022143212



PROJECT NUMBER : 6902  
PROJECT TITLE : Biochemical Special Investigations  
PROJECT LEADER : B. D. Davies  
PERIOD COVERED : July, 1991

## **I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY**

- A. **Objective:** Develop a nicotine monoclonal antibody based enzyme-linked immunosorbent assay (ELISA) for the determination of nicotine concentrations in samples of interest to PM.
- B. **Results:** Some preliminary experiments were conducted which indicate that a 2% solution of bovine serum albumin (BSA) can be substituted for the 1% gelatin solution currently being used for blocking non-specific binding sites (1). These experiments have also indicated that the sensitivity of the assay is increased when BSA is used to both block non-specific binding and to wash the plate.
- C. **Plans:** Complete the experiments using BSA and determine if it will be substituted for gelatin in future assays. Synthesize some succinylated hydroxyethylnicotine conjugated alkaline phosphatase and examine an alternate ELISA protocol.
- D. **Reference:**  
Dunn, R. Notebook No. 9049, pp. 60-68.

## **II. METHOPRENE ASSAY EVALUATION**

- A. **Objective:** Evaluate the EnviroGard<sup>TM</sup> methoprene assay kit.
- B. **Results:** Freshly prepared calibrator solutions were prepared by the manufacturer and sent to PM. These calibrators were used in an ELISA along with three tobacco samples containing methoprene at 2.1, 5.0 and 9.9 ppm. Using these calibrators, the ELISA determined concentrations of methoprene of those tobacco samples were 2.45, 5.77, and 8.61. The maximum absorbance and percent maximum absorbance (%Bo) for the 0, 1 and 10 ppm calibrator solutions from that assay were also within the suggested guidelines.
- C. **Plans:** Obtain the commercially available kits currently being prepared by the manufacturer which incorporate new calibration standards and utilize the methanol extraction protocol. Upon receipt, evaluate their effectiveness.
- D. **Reference:**  
Crockett, E. Notebook No. 9061, pp. 179-191.

2022143213

### III. ELECTROPHORETIC TECHNOLOGY

- A. **Objective:** To develop electrophoretic techniques in-house including isoelectric focusing (IEF) and 2-dimensional (2-D) electrophoresis.
- B. **Results:** Balb/C 3T3 cell extracts were fractionated on the small format 2-D system. Discrete protein spots were well separated and easily visible on the silver stained gels. This format is now available for fractionating complex plant protein samples.

An aliquot of batch PMT was fractionated on the small format 2-D system. A tight cluster of approximately 100 proteins was observed in the 20 to 70 kD region. Approximately 10-20 proteins were found in the 60 kD region.

Photoaffinity labeled phenylethanolamine N-methyltransferase ( $^3\text{H}$ -PET) was prepared and fractionated on the small format 2-D system. The autoradiograph showed the presence of one major cluster of related spots and one minor radiolabeled spot. The position of the major cluster on the autoradiograph corresponded to that of PET.

An ammonium sulfate purified PMT preparation was subjected to preparative IEF fractionation on the RF3. While over 70% of the enzyme activity was recovered, there was only a 2 fold enrichment in PMT activity. The peak PMT activity containing fractions were refractionated using a narrower pH gradient. While there was a skewing of the enzyme activity peak from the protein peak, it was not sufficient enough to conclude that the technique would be a useful purification step at this stage.

- C. **Plans:** Examine the effectiveness of RF3 preparative IEF fractionation on phenylsepharose-purified PMT.
- D. **Reference:**

Krauss, M. Notebook No. 9088, pp. 46-63.

### IV. BIOSENSOR TECHNOLOGY

- A. **Objective:** Develop biosensor technology in-house to allow the determination of the concentration of gas phase compounds of interest.
- B. **Results:** Three piezoelectric crystals, previously coated with protein A, were further coated with a nicotine specific monoclonal antibody (HA4) and incubated in a solution containing 1 mM nicotine. While the crystals frequencies paradoxically increased after HA4 coating, they did decrease after incubation with nicotine, indicating specific binding of nicotine to HA4. The average decrease in frequency was  $119 \pm 29$  Hz.

- C. Plans: To regenerate the crystals by removing the nicotine, HA4 and protein A layers, repeat the coating procedures and expose the HA4 activated crystals to solutions of varying nicotine concentrations. From these studies determine the dose dependent relationship between nicotine concentration and decreasing frequency (Hz).

D. Reference:

Crockett, E. Notebook No. 9061, pp. 179-191.

2022143215

PROJECT NUMBER : 6906  
PROJECT TITLE : Biological Effects of Smoke  
PROJECT LEADER : G. M. Nixon  
PERIOD COVERED : July, 1991

**I. SALMONELLA/MICROSOME (S/M) ASSAY (D. Stagg)**

- A. **Objective:** To test the biological activity of experimental CSCs and other pertinent materials.
- B. **Results:** Twenty-eight CSCs were tested this month in support of the Cross Soluble Base Web studies. Also two pure compounds were analyzed.
- C. **Plans:** Complete ongoing studies and document all results in appropriate memos. Continue to test samples for biological activity as they become available. Continue to evaluate the "screening" assay protocol as it is currently being performed.
- D. **References:**
- Jones, R. Notebook No. 8769, p. 141.
- Stagg, D. Notebook No. 9110, p. 125.

**II. 3T3 CELL BIOCHEMICAL STUDIES (G. Nixon)**

- A. **Objective:** To investigate the induction of (putative) heme oxygenase (HO) in 3T3 cells under various conditions.
- B. **Results:** 3T3 whole cell extraction experiments were performed in a continuing investigation of the induction of (putative) HO. Experiments testing the effects of buthionine sulfoximine (BSO) pretreatment followed by 12-O-tetradecanoylphorbol-13-acetate (TPA) or sodium arsenite treatment were repeated using both a 2-hour [35S] methionine labeling period as in previous experiments, and a 30-minute labeling period. For the longer labeling period, results were similar for both TPA and arsenite. For the shorter labeling period, there were dramatic differences between the two compounds. TPA treatment resulted in a rapid increase in a 32kD protein (putative HO), with a corresponding decrease in actin levels. The response to arsenite in the short label protocol was minimal. Cycloheximide (CHX) was used to inhibit protein synthesis in two different protocols using a short labeling period. Results paralleled those seen for previous experiments using a two-hour labeling period. Finally, an experiment testing the effects of several inhibitors of protein kinase C was repeated using the short and long labeling periods. Results are not yet available for this experiment.
- C. **Plans:** 3T12 cells (transformed 3T3 cells) will be tested for HO induction using the same experimental protocols which were used for 3T3 cells.

2022143216

**D. References:**

Burruss, T. J. Notebook No. 8896, pp. 38-39.

Nixon, G. M. Notebook No. 8711, pp. 173-174.

2022143217

PROJECT NUMBER : 6908  
PROJECT TITLE : Smoke Condensate Studies  
PROJECT LEADER : A. H. Warfield  
WRITTEN BY : W. R. Morgan  
PERIOD COVERED : July, 1991

**I. REDUCTION OF MS TSNA BY REDUCING PREFORMED TSNA AND PYROSYNTHETIC TSNA PRECURSORS IN FILLER**

- A. **Objective:** Reduce MS TSNA by selective removal of TSNA, amine precursors, and/or nitrosating agent precursors from filler.
- B. **Results:** MS smoke TSNA values for the low TSNA blend model cigarette were obtained. Filler data for the same blend was reported in the June monthly summary. The MS data show reductions of 62%, 59% and 5% for NNN, NAT and NNK respectively.
- C. **Plans:** Continue development of a low TSNA prototype with particular emphasis on reduction of NNK. Prepare a completion report on the first phase of the development of the low TSNA blend model.
- D. **Reference:**  
Tickle, M. H. Notebook No. 9096, pp. 81-82.

**II. REDUCTION OF MS TSNA BY INHIBITING THE PYROSYNTHESIS OF TSNA**

- A. **Objective:** Reduce the levels of pyrosynthesized MS TSNA by blocking reaction pathways which form TSNA.
- B. **Results:** SCFE oriental sub-fractions 2 and 5 were applied to SCFE Bu to assess the effect on MS delivery of TSNA. There was no significant reduction of TSNA delivery in MS smoke from either fraction. The cumulative results from the oriental sub-fraction inhibitor study show that only sub-fraction 0 (benzene solubles) shows any propensity toward reduction of MS TSNA. Sub-fraction 0 yielded reductions of 42%, 73% and 39% for NNN, NAT and NNK respectively. Further fractionation of the oriental SCF extracts is not planned.
- C. **Plans:** Initiate a study directed at defining the mechanism of reduction of TSNA from oriental sub-fraction 0.
- D. **Reference:**  
Haut, S. A. Notebook No. 9078, pp. 39-40.

2022143218

### III. UNEXTRACTED NICOTINE STUDIES

- A. **Objective:** To determine whether there is a correlation between unextracted nicotine in filler and the delivery of NNK in the corresponding MS smoke.
- B. **Results:** Experiments to determine the stability of TSNA under the basic digestion procedure used to analyze for NIC-X and NIC-Y were conducted. Digestions of 18 hours using various temperatures (Refrigerated (0°C), Room Temperature (22°C), 30°C and 40°C) and NaOH concentrations (0.01, 0.05, 0.1 and 0.5 N) were made using TSNA standard solutions. NNN was relatively immune to degradation under all temperatures and concentrations studied. NAT was degraded at all temperatures and NaOH concentrations with the greatest amount of degradation occurring at the highest temperatures and concentrations. NAT levels of destruction ranged from 50% at 0°C and 0.5 N to 100% at 40°C and 0.5 N NaOH. NNK was degraded as much as 40% at 0.5 N NaOH at 0°C but at lower NaOH concentrations 80 - 90% was recovered regardless of the temperature. These results indicate that NNN and NNK results can be obtained from the digestion process with a 0.1 N NaOH concentration at any of the temperatures studied. NAT results can only be obtained at the lowest temperatures and NaOH concentrations.
- C. **Plans:** Investigate the stability of TSNA in tobacco samples when subjected to the above digestion processes.
- D. **Reference:**

Lambert, E. A. Notebook No. 9108, pp. 34-36.

### IV. ANALYTICAL PROCEDURES

- A. **Objective:** To develop and maintain analytical methodology for TSNA or other compound classes where information is needed for determining relationships of TSNA to their precursors.
- B. **Results:** Work continued on development of a method for nitrite and nitrate in filler using ion chromatography (IC). The chloride interference with the analysis of nitrite was effectively solved by passing the aqueous tobacco extract through a sulfonic acid SPE column which had been pretreated with silver nitrate. This procedure removes chloride from the tobacco extract solution thus allowing for analysis of nitrite. The nitrite/nitrate method was also tested with lower level standards and increased sensitivity. Standards containing 0.015 µg nitrite and 0.12 µg nitrate per ml of solution were easily detectable. Working limits of 0.1 µg nitrite nitrogen and 1.0 µg nitrate nitrogen per gram of tobacco have been established.
- C. **Plans:** A series of 15-20 fillers is now being analyzed by this method. Included are many water washed fillers, some base webs and some control fillers.

**D. Reference:**

Warfield, A. H. Notebook No. 8847, pp. 83-85.

**V. MISCELLANEOUS AND SUPPORT STUDIES**

- A. Objective:** To conduct studies of the TSNA content of filler and/or smoke and carry out other activities as necessary to support other PM programs.
- B. Results:** Studies being conducted in conjunction with Dr. F. Hsu (ARD) relating to the identification of individual nitrosamines in smoke continued this month. The GC/MS and GC/TEA operating parameters have been established and analysis of smoke samples has begun. The confirmation of NNK in mainstream smoke from BuBW and aged sidestream smoke was a first priority. It was of interest to isolate and identify the component in each of these two cases which gives rise to the observed TEA response at a retention time equal to that in a standard for NNK. The results show that for both BuBW and aged SS NNK is the component producing the TEA response.
- C. Plans:** Further studies are planed to investigate the origin of NNK in BuBW. Studies are also planed to use the GC/MS to investigate other TEA responses to identify the component producing the response.
- D. Reference:**
- Morgan, W. R. Notebook No. 8905, p. 36.

2022143220



PROJECT NUMBER : 6912  
PROJECT TITLE : Tobacco/Smoke Relationships  
PROJECT LEADER : R. R. Izac  
PERIOD COVERED : July, 1991

## **I. CROSS SOLUBLES BASE WEB (CHEMISTRY)**

A. **Objective:** To modify tobacco solubles and evaluate the smoke chemistry of fillers from various treated solubles and the appropriate base web.

B. **Results:** The BuS1 samples treated with silver nitrate, silver acetate or silver carbonate were sprayed onto filler and model cigarettes prepared. CSC collected from these cigarettes have been submitted for biological and analytical analysis.

Bu11 has been treated with pectinase, pronase and a mixture of pectinase and pronase. Model cigarettes were generated from the insoluble residue and the solubles. CSC from these model cigarettes were submitted for the S/M assay.

In collaboration with the personnel of Project 1902, an evaluation of the microbial fermentation of BuS1 has begun.

CSC from handmade and machine made cigarettes were submitted to the S/M assay for use as controls.

C. **Plans:** Continue to generate model fillers from various CSBW studies. Submit IT CSCs from various studies for S/M testing.

D. **References:**

1. Zoller, M. Notebook No. 9045, pp. 77-84.
2. Hassam, S. Notebook No. 9114, pp. 6-33.
3. Drew S. Notebook No. 9057, pp. 86-87 and 94.

## **II. SUPPORT FUNCTION: SAMPLE PREPARATION**

A. **Objective:** To prepare chemical fractions and/or condensates as needed for biological and chemical analysis.

B. **Results:** About 85 samples from various sources were assayed via HPLC for nicotine. A sample of nicotine succinate was partially purified using HPLC for the personnel of Project 6902. Three samples obtained from the PMT assay were analyzed for amines by HPLC. These samples were submitted to ARD for analysis. About 499 cigarettes have been prepared for model development and about 117 samples have been smoked for special projects. Single smokings of 2R1 cigarettes using different trapping methods were made to collect smoke for GC smoke profile analysis.

2022143221

**C. References:**

1. Izac, R. R. Notebook No. 8874, pp. 121-123.
2. Zoller, M. Notebook No. 9045, pp. 77-84.
3. McGee, N. Notebook No. 9104, pp. 16-30.
4. Hellams, R. Notebook No. 8959, p. 189.

2022143222

PROJECT NUMBER: 8101  
PROJECT TITLE : Cigarette Testing Services  
WRITTEN BY : Kathleen A. Mitchell  
PERIOD COVERED : July, 1991

## **I. MARKET ACTIVITY**

**A. Objective:** To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

**B. Results:**

**Domestic - New Brands**

R. J. Reynolds is test marketing Camel Ninety99Nines 100 (Box) (18 mg tar, 1.3 mg nicotine) and Camel Lights Ninety99Nines 100 (Box) cigarettes (10 mg tar, 0.8 mg nicotine). These are box line extensions of the Camel Full Flavor and Lights versions. These cigarettes were picked up in California, although the geographic extent of this introduction is still undetermined.

Brown & Williamson is nationally distributing Raleigh Extra Non-Filter (24 mg tar, 1.3 mg nicotine) cigarettes at sub-generic prices.

Lorillard is test marketing True Long Size (Box) (5 mg tar, 0.4 mg nicotine) and True 100 (Box) (6 mg tar, 0.5 mg nicotine) cigarettes. The test market areas designated for this product are predominately strong True and box category markets. These cigarettes were received from Maine and New Hampshire.

Lorillard is test marketing Old Gold 100 (19 mg tar, 1.4 mg nicotine) and Old Gold Lights 100 (13 mg tar, 1.0 mg nicotine) as branded generics in Kentucky. During June, Old Gold will be tested in Montana as a branded generic. Old Gold is still being sold as a full margin product in all other locations. These products are identical in packaging to the full margin products already on the market.

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